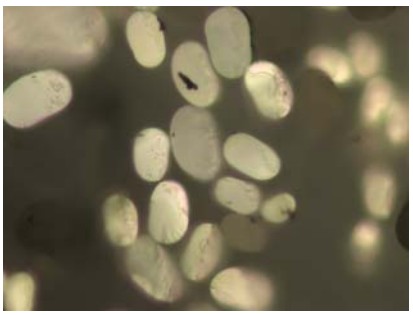
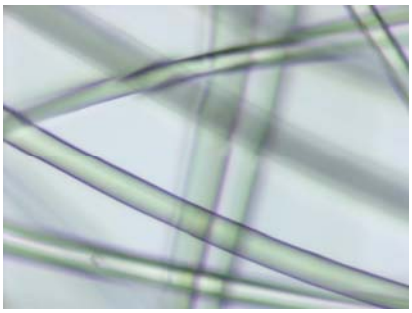
JRC Scientific and Technical Reports

Fibre Labelling
Melamine - Basofil

FINAL REPORT
Administrative Arrangement N. 2006-30035
Analysis conducted on behalf of DG ENTERPRISE

P. Piccinini, E. Buriova, C. Senaldi, S. Yazgan





EUR 23482 EN - 2008

JRC EUROPEAN COMMISSION

ihcp Institute for Health and Consumer Protection

The mission of the IHCP is to provide scientific support to the development and implementation of EU policies related to health and consumer protection.

The IHCP carries out research to improve the understanding of potential health risks posed by chemical, physical and biological agents from various sources to which consumers are exposed.

European Commission
Joint Research Centre
Institute for Health and Consumer Protection

Contact information
Address: Paola Piccinini, T.P. 260, via E. Fermi 1, Ispra (VA), 21020, Italy
E-mail: paola.piccinini@jrc.it
Tel.: 0039-0332-789124
Fax: 0039-0332-785707

http://ihcp.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/

JRC 46848

EUR 23482 EN
ISBN 978-92-79-09577-1
ISSN 1018-5593
DOI 10.2788/91012

Luxembourg: Office for Official Publications of the European Communities

Printed in Italy

TABLE OF CONTENTS

1. EXECUTIVE SUMMARY……..1

2. INTRODUCTION……5

3. TEST METHODS FOR IDENTIFICATION, QUANTIFICATION AND CHARACTERISATION OF THE NEW FIBRE……..7

3.1 BACKGROUND……..……7

3.2 IDENTIFICATION METHODS……..….9

3.2.1 Microscopy.…….9

3.2.2 FT-IR.…….10

3.2.3 Thermo gravimetric analysis.…….11

3.2.4 Differential scanning calorimetry.…….13

3.3 QUANTIFICATION METHODS……..…….14

3.3.1 Pre-treatment.…….14

3.3.2 Agreed allowance.…….15

3.3.3 Chemical methods.…….17

3.3.4 Methods proposed by the applicant.…….19

3.3.5 Results.…….20

4. CONCLUSIONS…….39

5. REFERENCES……...43

6. ANNEX I – ANALYTICAL METHODS PROPOSED BY THE APPLICANT……...45

7. ANNEX II – MICROSCOPIC ANALYSIS…….59

8. ANNEX III – SPECTROSCOPIC ANALYSIS……...67

9. ANNEX IV – THERMOGRAVIMETRIC ANALYSIS…….79

10. ANNEX V – ANALYSIS OF COMPOSITION……...91

AGREED ALLOWANCE.....…...93

PRE-REATMENT.....…...97

CORRECTION FACTOR *d* ACCORDING TO DIRECTIVE 96/73/EC…..……...…103
CORRECTION FACTOR *d* ACCORDING TO METHODS PROPOSED BY THE APPLICANT……..……114
CHEMICAL QUANTIFICATION……...…...……..127
INFLUENCE OF FORMIC ACID CONCENTRATION……...……...……...152
INFLUENCE OF TEMPERATURE ON HOT SULPHURIC ACID METHOD.....154
INFLUENCE OF TEMPERATURE ON HOT FORMIC ACID METHOD…….....156
INFLUENCE OF TEMPERATURE ON HOT SODIUM HYPOCHLORIDE METHOD …….....162
INFLUENCE OF TEMPERATURE ON METHOD 2 OF DIRECTIVE 96/73/EC.....164
INFLUENCE OF TEMPERATURE ON METHOD 7 OF DIRECTIVE 96/73/EC.....165

1. Executive Summary

In 2006, the European Commission received a petition, presented by Basofil Fibers LLC, for the establishment of a new generic name, in accordance with Directive 96/74/EC. The proposed name was melamine fibre and the suggested definition is reported as follows: “fibre formed of at least 50 % by mass of cross-linked macromolecules made up of melamine polymer”.

The Commission therefore convened two meetings of the technical working group for Directive 96/74/EC on textile names, comprising governmental experts representing each Member State. The meetings were held in Brussels on 6th February and 15th May 2006. The application was considered to be justified by the group of experts, who recommended an amendment to the list of fibre names in Annex I of Directive 96/74/EC. As a result of several discussions with technical experts, the name proposed by the Commission for the new fibre is melamine and it will be thus indicated for the purpose of this report.

In August 2006, the European Commission’s Joint Research Centre (JRC) was asked to conduct experimental work to check the validity and suitability of the testing methods proposed by the applicant for the identification, quantification and characterisation of melamine.

The results of this investigation were presented during the seventh and eighth technical meeting of the European network of national experts on textile labelling, held in Ispra on 19th September 2007 and 21st April 2008.

The tests performed by the JRC and described in this report confirmed that test methods are available for the identification, quantification and characterisation of the new fibre melamine.

Although the new fibre has quite a characteristic distribution of varying elliptical cross-sections, experimental results showed that microscopic analysis can be useful but it is not the only technique for the identification of melamine as it can give rise to mistakes in the identification of man-made fibres. Identification should be based on FT-IR, possibly combined with Thermo Gravimetric (TG) analysis and solubility properties of the fibre.

The agreed allowance of melamine fibre was experimentally evaluated and the value of 7.00 was approved during the meeting of the European network of national experts on textile labelling held in Ispra on 21st April 2008.

The normal pre-treatment, described in Directive 96/73/EC on certain methods for the quantitative analysis of binary textile fibre mixtures, was shown to be applicable to the new fibre. Both traditional Soxhlet and automatic extractor (Soxtec) can be used to this purpose.

Melamine is insoluble with all methods of Directive 96/73/EC. The correction factors *d* for all the chemical methods described in the Directive (except method 12) were calculated: values were generally equal to 1.01, except in the case of method 3 and 10, for which *d* value was 1.00. As agreed on 19th September 2007 during the 7th meeting of the European network of national experts on textile labelling, five correction factors *d* of melamine were confirmed through a ring trial including seventeen laboratories. The inter-laboratory trial results established the value of 1.01 as melamine correction factor for methods 3, 4, 8 and 14. The *d* value calculated in the ring trial for method 7 was 1.07, however the repeatability and reproducibility limits calculated were very poor and the data could not be considered as being part of a normal distribution. Consequently, at the 8th meeting of the Network on 21th April 2008, it was decided that method 7 would not be considered applicable to blends containing melamine.

Three methods were proposed by the applicant and tested by the JRC for quantification purposes: the first one makes use of a 6.15 % sodium hypochloride solution to dissolve cellulosic fibres, the second one of a 50 % sulphuric acid solution to dissolve melamine and the third one of a 90 % formic acid solution to dissolve melamine. All the proposed methods foresaw a temperature of 90 °C and a contact time of three hours.

For the three methods, the solubility properties of melamine and other common fibres, supposed to be found in mixture with melamine, were evaluated, together with the effect of key parameters, such as contact time, temperature and concentration of reagent. In addition, the performance of the two methods using hot acid solutions in the quantification of pure melamine and binary blends with aramid was compared.

Based on the experimental data, the following conclusions were agreed with experts from Member States, concerning the three methods proposed by Basofil.

The method with sodium hypochloride at 90°C was deemed not applicable due to partial solubility of melamine, which was supposed to be insoluble.

The hot formic acid method was considered adequate and applicable in the case of binary mixtures of melamine with aramid and cotton. It was decided that the contact

time could be reduced to one hour, as no difference in quantitative results of mixtures and pure melamine were obtained when one hour was used instead of three as contact time. The temperature proved to have a strong influence on this method and it should be carefully maintained in the range 90 ± 2 °C for the whole period of contact time.

The hot sulphuric acid method was judged not adequate as results obtained with this method were statistically different compared to those obtained with the hot formic acid method and the reagent shown difficulties in dissolving completely melamine.

During the 7th meeting of the European network of national experts on textile labelling, a consensus was reached on the need to validate the new method based on hot formic acid and to confirm the correction factors *d* for aramid (meta and para) and cotton. The JRC organised the ring trial with the participation of seventeen European laboratories, in accordance with the rules laid down in ISO 5725 (1994), and discussed results during the 8th meeting of the network. The validation was successful. The precision of the test method, which should be quoted as a percentage by mass, was expressed as repeatability and reproducibility limits. Results were 0.74 % and 1.77 %, respectively. The correction factors *d* for aramid and cotton were evaluated in the same context and the established values are 1.02 for both fibres.

On the basis of the experimental results and of discussions with representative experts from Member States (meetings on 6th February, 15th May, 28th June, 19th September 2007 and 21st April 2008), the definition agreed and proposed for melamine is: "*fibre formed of at least 85 % by mass of cross-linked macromolecules made up of melamine derivatives*".

The name melamine was chosen, in agreement with experts from Member States, as it fulfils the criteria set up in 2002 by Commission and technical experts working group on textile labelling. In fact, according to these criteria, a generic name should not link the fibre to a specific manufacturer, it should be free of rights and it should inform consumers about characteristics of the fibre.

2. Introduction

In 2006, the company Basofil Fibers asked the European Commission to establish a new generic fibre name under Directive 96/74/EC on textile names [1], as they claimed the novelty of their new fibre. As a result of several discussions with technical experts, the name proposed by the Commission for the new fibre is melamine and it will be thus indicated for the purpose of this report.[1]

Melamine is a fibre made from a condensation polymer of melamine, melamine derivatives and formaldehyde-supplying products. In the condensation reaction, methylol compounds are formed which then react with one another to form a three-dimensional structure of methylene (-CH_2-) and dimethylene ether (-CH_2-O-CH_2-) bridges. Melamine monomeric derivatives have the following generical formula:

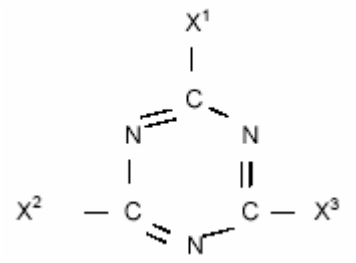


Fig. 1: Chemical structure of melamine.

Where X_1, X_2 and X_3 can be $–NH_2$, -NHR_1 or $–NR_1R_2$, although X_1, X_2 and X_3 must not all be $–NH_2$. R_1 and R_2 can be HO-C_2-C_{10}-alkyl, HO-C_2-C_4-alkyl-(oxa-C_2-C_4-alkyl)$_n$ or H_2N-C_2-C_{12}-alkyl. Melamine is a heat and flame resistant fibre with low thermal conductivity. It was developed by BASF AG in the early 1990's. Melamine is produced as a staple fibre and is almost always combined with other natural and/or synthetic fibres (in most cases it is mixed with aramid, cotton, modacrylic, polyester and viscose). Due to its heat and flame resistance, melamine is mainly used in fire blocking fabrics, protective clothing and high temperature filtration products. The petition was firstly discussed on 6th February 2006 in Brussels during a meeting of the technical expert working group on textile labelling, composed of Member States' governmental experts associated with the Committee for Directives relating to Textile

1. The International Bureau for the standardisation of man-made fibres (BISFA) and the USA's Federal Trade Commission (FTC) have already established the generic name "melamine".

Names and Labelling. Based on the following agreed set of criteria, the group of experts considered that the petition was justified:

1. the new fibre should be radically different from other fibres by chemical composition and/or by manufacturing route and production process;
2. fibre characteristics can be taken into account but need to be examined on a case by case basis;
3. the new fibre should be detectable and distinguishable from other fibres by standardised test methods;
4. consumer relevance should be shown by active commercial use of the fibre;
5. a new name is justified only if the fibre cannot be classified into existing groups.

The group judged that experimental work was needed to verify the applicability of the proposed analytical methods for identifying and quantifying melamine in blends (see Annex I). In fact, validated test methods, enabling market surveillance authorities in Member States to determine the composition of textile products containing the new fibre, should be established at European level. An amendment to Directive 96/74/EC on textile names [1] would subsequently be prepared.

Within the framework of the Commission's investigation on this fibre, the Joint Research Centre (JRC) was charged by DG Enterprise with the analytical work and, in particular, with the verification of the validity and suitability of the test methods proposed by the petitioner.

3. Test methods for identification, quantification and characterisation of the new fibre

3.1 Background

The experimental work was discussed and agreed with national technical experts, representing Member States, during meetings held in Brussels on 6th February 2006 and 15th May 2006 and in Ispra on 28th June 2006, 19th September 2007 and 21st April 2008. It was decided that the JRC would work on pre-treatment, determination of agreed allowance and correction factors *d*, verification of identification and quantitative methods (based on chemical dissolution).

The JRC selected relevant samples, in collaboration with the applicant, taking into account the market for melamine and the possible range of compositions in blends. Melamine can be used in woven or knitted fabrics, typically in binary mixtures with aramid (meta and para), cotton, modacrylic, polyester and viscose. Based on this market analysis, fabric samples made by binary and ternary mixtures containing melamine in various percentages were considered for the experimental phase.

Table 1 show samples received from Basofil and analysed during this project. Samples **136**,**171** and **179** are staple fibres of pure melamine from bobbin. Samples **137** and **180** are needle-punched felts of pure melamine. Samples **138** to **143** and **177-178** are spun yarns or fabric samples of mixtures of melamine and aramid (meta and para). Samples **150** to **154** are staple fibres from bobbin of pure fibres. Sample **144** is a ternary mixture of melamine and polyester/modacrylic. Sample **145** is a quaternary mixture of polyester/modacrylic/lyocell and melamine. All the other samples are binary mixtures, either woven or non woven fabrics.

Table 1: Samples received from Basofil.

JRC code	Composition	Sample type	Color
136	100 % MLF (Merge77)	staple fiber	white
137	100 % MLF	needle-punched felt	white
138	40 % MLF - 60 % m-aramid (Nomex)	knitted hood	white
139	40 % MLF - 60 % m-aramid (Nomex)	woven fabric	blue
140	50 % MLF - 50 % m-aramid (Nomex 462)	spun yarn	white
141	40 % MLF - 60 % p-aramid (Kevlar)	spun yarn	yellow
142	40 % MLF - 60 % p-aramid (Kevlar)	ripstop woven fabric	yellow
143	25 % MLF - 75 % m-aramid (Conex)	felt	brown
144	17 % MLF - 17 % modacrylic - 66 % polyester	woven fabric	white
145	12 % MLF - 30 % lyocell - 34 % modacrylic - 24 % lowmelt polyester	flame resistant gighloft barrier nonwoven	white
150	100 % p-aramid (Kevlar)	staple fibre	yellow
151	100 % m-aramid (Nomex)	staple fibre	white
152	100 % lyocell (Tencel)	staple fibre	white
153	100 % lowmelt polyester	staple fibre	white
154	100% modacrylic	staple fibre	white
171	100% MLF (Merge 80)	staple fibre	slightly yellow
172	MLF/viscose	fabric apparel	dark grey
173	MLF/Visil (quilted to FR Cotton shell fabric)	termal liner	grey
174	MLF/polyester	knit fabric	white
175	MLF/cotton	fabric apparel	grey
176	MLF/FR viscose	innerliner fabric	white
177 = 139	MLF/m-aramid (Nomex)	fabric apparel	blue
178 = 142	MLF/p-aramid (Kevlar)	fabric turnout	yellow
179	100 % MLF	staple fibre	white/yellow
180	100 % MLF	needle-punched felt	yellow

Table 2: Other samples used during this project.

JRC code	Composition	Sample type	Color
005	100% cotton	knitted sock	white
034	100% viscose	knitted sock	white
038	100% polyamide	knitted sock	white
040	100% triacetate	knitted sock	white
057	100% p-aramid (Kevlar)	yarn from bobbin	yellow
159	100% cotton	fabric	white
181	100% cotton	yarn from bobbin	sligtly yellow
182	100% p-aramid (Twaron)	staple fibre	yellow
183	100% m-aramid (Conex)	staple fibre	blue
222	100% m-aramid (Conex)	yarn from bobbin	white

Note 1: In this and in other tables MLF stands for melamine.

Note 2: Visil is a flame retardant form of viscose which has silica embedded in the fibre during manufacturing.

3.2 Identification methods

The method proposed by the applicant for identifying melamine is based on microscopic analysis and on Fourier transform infrared spectroscopy (FT-IR) (see Annex I). In this section results obtained with these techniques plus thermo gravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC) are reported. Complete results are shown in Annex II, III and IV. Solubility properties can help in identifying melamine, as this fibre is insoluble in several solvents and aqueous solution, as described in chapter 3.3.5.

3.2.1 Microscopy

Even though melamine has a unique distribution of varying elliptical cross-sections, it cannot be easily and unambiguously identified by microscopic analysis, as evident from the photos of longitudinal and cross-section analysis reported in the following, as well as in Annex II.

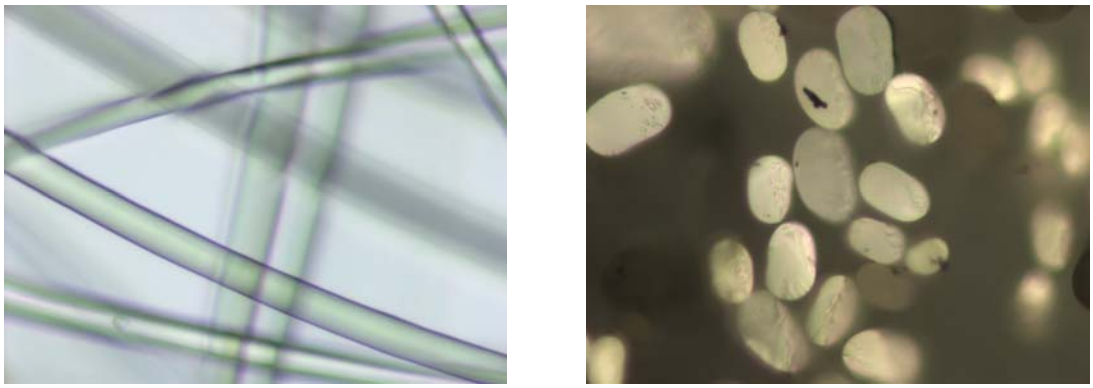


Fig. 2: Pure melamine (sample **136**). **a)** Longitudinal view, 630X; **b)** Cross section, 630X.

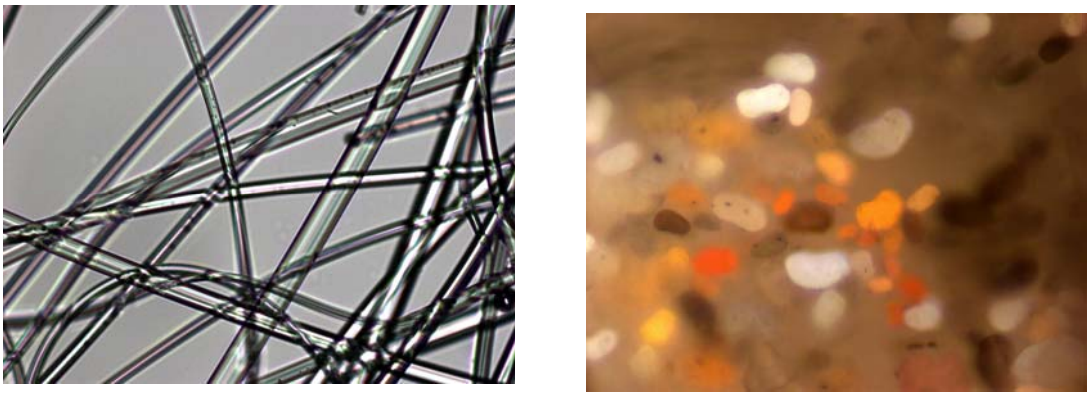


Fig. 3: Pure melamine (sample 137). a) Longitudinal view, 200X; b) Cross section, 400X.

Nowadays the cross-section distribution range of melamine varies from 5 to 25 µm with an average of 15 µm. Microscopic analysis can be used as an identification technique only if combined with other identification techniques such as FT-IR spectroscopy or thermo gravimetric analysis.

All samples received from Basofil were analysed by microscopy for a preliminary characterisation and photos are reported in Annex II. A Zeiss microscope model Axioskop 2 Mat was used and analyses were performed using transmitted light. Glycerol triacetate was used as contrast reagent.

3.2.2 FT-IR

The nature of the new fibre can be proved by means of Fourier transform infrared spectroscopy (FT-IR). In the comparison of the obtained reflectance spectrum with the known spectrum of pure melamine a quality match of 75 % or greater is required, as a criterion of judgement, to confirm the presence of melamine in the sample. Recognition of melamine is easy due to the fingerprint of the molecule and to some characteristic peaks due to the chemical structure of the fibre, such as for example methylene (-CH_2-) and dimethylene ether (-CH_2-O-CH_2-) bridges and alcohol or amine groups.

Figure 4 shows the FTIR spectrum of untreated melamine.

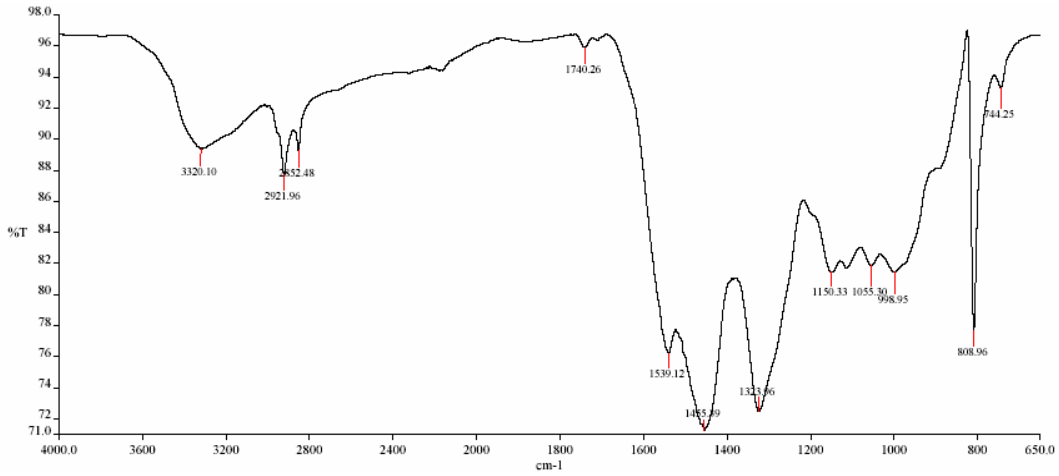


Fig. 4: FT-IT spectrum (ATR) of untreated melamine (sample **137**).

FT-IR spectra of all samples were acquired using ATR Attenuated Total Reflectance mode with a Perkin Elmer instrument (FT-IR spectrometer spectrum 2000). Samples were analysed without any preparation.

Fabric made of binary and ternary mixtures were also analysed and their spectra were compared to the ones of pure fibres. All spectra of pure fibres and fabric samples are reported in Annex III.

3.2.3 Thermo gravimetric analysis

Experiments on thermo gravimetric analysis were also performed in the JRC's laboratories in order to investigate the suitability of this technique as an identification method for melamine. The thermogram of pure melamine is reported in Figure 5.

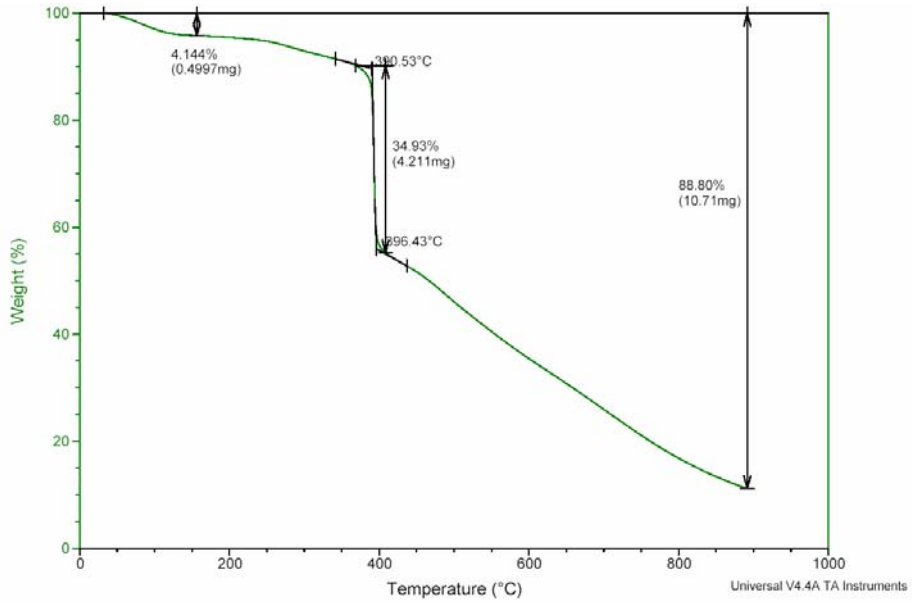


Fig. 5: TGA analysis of pure melamine (sample **136**).

Thermo gravimetric analysis can be used as identification technique only in some cases. When melamine is blended with fibres such as polyester, cotton and viscose, it is difficult to recognize its presence from the thermogram. On the contrary thermogravimetric analysis of binary mixtures with aramid, in particular p-aramid, allows the identification of the two fibres, as reported in Figure 6, as well as in Annex IV.

Table 3 reports the range of temperature in which the inflection points of common fibres, known to be used in blends with melamine, can be found. Even if TG analysis cannot be applied as an absolute method to identify melamine from other fibres, it can be used combined with other techniques such as FT-IR spectroscopy and microscopic analysis as a confirmative analysis in the identification of melamine.

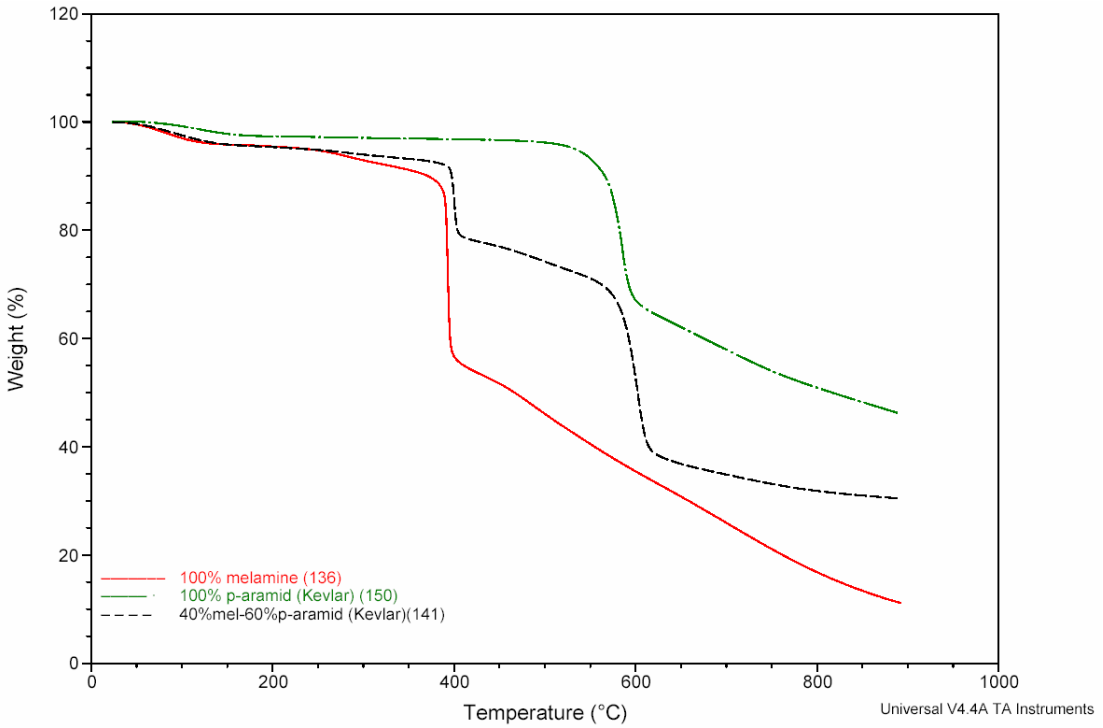


Fig. 6: TGA analysis of pure melamine (sample **136**), pure p-aramid (Kevlar) (sample **150**) and a binary mixture melamine/ p-aramid (Kevlar) (sample **141**).

Table 4: Inflection points of pure fibres used in mixtures with melamine.

	Inflection Point Temperature (°C)					
	100-200	**200-300**	**300-400**	**400-500**	**500-600**	**600-700**
m-aramid (Conex)				431-461		
m-aramid (Nomex)				454-466		
p-aramid (Twaron)					587	
p-aramid (Kevlar)					582-601	
cotton			355-379			
melamine			**387-400**			
modacrylic		247-275				
polyester				441-446		
lyocell (Tencel)			360			
viscose			357-369			

The equipment used for the analyses was a TGA model Q500 by TA Instruments. A temperature program of 20 °C/min, starting from 25 °C up to 900 °C, was set with a nitrogen gas flow of 60 ml/min. Sample weight was in the range 1-10 mg.

Binary and ternary mixtures samples were also analysed and their thermograms were compared to the ones of pure fibres. All thermograms of pure fibres and fabric samples are reported in Annex IV.

3.2.4 Differential scanning calorimetry

Analyses made by Differential Scanning Calorimetry (DSC) were also performed. However results showed two peaks too broad to be used in the identification of the fibre, as reported in Figure 7. In fact the first peak starts at about 83 °C and finishes at 154 °C (with a maximum at 120 °C), while the second one starts at 218 °C and ends at 278 °C (with a maximum at 250 °C).

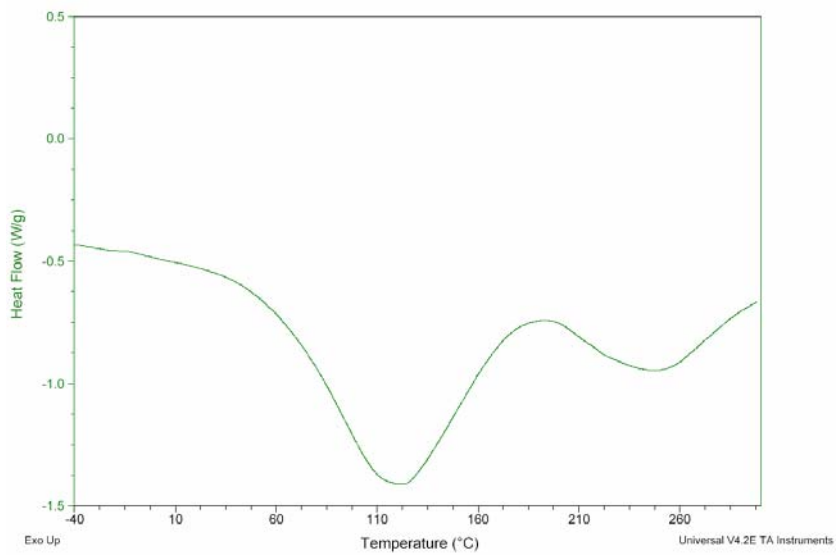


Fig. 7: DSC analysis of pure melamine (sample **136**).

The equipment used for the analyses was a DSC model Q100 by TA Instruments. A temperature program of 20 °C/min, starting from -40 °C up to 300 °C, with a nitrogen gas flow of 50 ml/min was employed. Sample weight was in the range 1-10 mg.

3.3 Quantification methods

The JRC quantitatively analysed all the samples received by Basofil (listed in Table 1) by chemical analysis as described in Directive 96/73/EC [2] and as described in the applicant's petition (see Annex I). Unfortunately quantification based on manual separation was impossible, due to the fact that melamine is produced as staple fibre and mixed with other fibre to prepare yarns, so that each yarn in fabrics is made by an intimate mixture of fibres.

3.3.1 Pre-treatment

Before quantification, samples should be pre-treated, in order to eliminate non-fibrous matter. Directive 96/73/EC [2] suggests extracting non-fibrous matter with light petroleum ether and water. The procedure foresees a one-hour extraction in Soxhlet with light petroleum ether (boiling range 40 to 60 °C), followed by a one-hour extraction in water at room temperature and a one-hour extraction in water at 65 ± 5 ºC, using a liquor/specimen ratio of 100/1. Results on samples of pure melamine, showed mass loss of about 0.70 % (Table 5). This value was in line with what stated by the producer regarding the content of spin finishing agent usually found on melamine. Some pre-treatments were performed using an automatic hot-extractor (Soxtec) instead of traditional Soxhlet, no differences in terms of mass loss were noticed.

Table 5: Mass loss of pure melamine due to pre-treatment.

JRC code	composition	pre-treatment	replicates	mass loss %	unc. at 95 %	RSD %
136	100 % melamine	Soxhlet	16	**0.70**	**0.06**	14.88
136	100 % melamine	Soxtec	15	**0.70**	**0.07**	18.22
137	100 % melamine	Soxhlet	10	**0.75**	**0.08**	15.29
137	100 % melamine	Soxtec	10	**0.74**	**0.11**	19.87
171	100 % melamine	Soxtec	3	**0.78**	**0.22**	11.55
179	100 % melamine	Soxtec	6	**0.92**	**0.04**	4.63

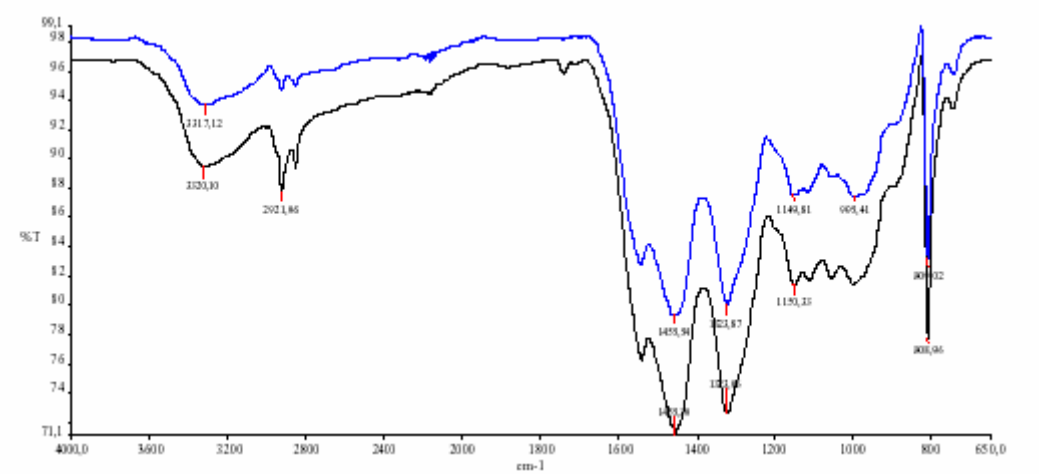


Fig. 8: FT-IT spectra (ATR) of pure untreated (---) and pre-treated (---) melamine **136** (Soxhlet).

Figure 8 shows the comparison of FTIR spectra of melamine (sample **136**) as received and after pre-treatment.

The pre-treatment of binary, ternary and quaternary mixtures was also performed and the values of mass loss obtained were generally in the range of 0.5 to 1.2 %, as shown by results reported in Table 6.

The detailed results are reported in Annex V.

Table 6: Mass loss of fabric samples due to pre-treatment.

JRC code	stated composition	pre-treatment	replicates	mass loss %	unc. at 95 %	RSD %
138	40 % MLF - 60 % m-aramid (Nomex)	Soxhlet	3	**0.82**	**0.12**	5.97
139	40 % MLF - 60 % m-aramid (Nomex)	Soxhlet	3	**0.73**	**0.09**	5.21
140	50 % MLF - 50 % m-aramid (Nomex) 462	Soxhlet	5	**0.25**	**0.01**	3.31
141	40 % MLF - 60 % p-aramid (Kevlar)	Soxhlet	3	**0.61**	**0.11**	7.26
142	40 % MLF - 60 % p-aramid (Kevlar)	Soxhlet	3	**0.71**	**0.10**	5.86
143	25 % MLF - 75 % m-aranid (Conex)	Soxhlet	3	**0.52**	**0.10**	7.90
144	17 % MLF - 17 % modacrylic - 66 % PES	Soxhlet	5	**0.28**	**0.03**	7.66
145	12% MLF - 30% lyocell - 34% modacrylic 24% Lm PES	Soxhlet	3	**0.46**	**0.12**	10.40
172	Basofil - FR viscose	Soxtec	3	**0.81**	**0.08**	4.13
173	Basofil - Visil	Soxtec	3	**0.97**	**0.03**	1.23
174	Basofil - polyester	Soxtec	3	**0.69**	**0.09**	5.15
175	Basofil - cotton	Soxtec	3	**0.86**	**0.02**	0.72
176	Basofil - FR viscose	Soxtec	3	**0.62**	**0.12**	7.61
177	Basofil - m-aramid (Nomex)	Soxtec	3	**1.16**	**0.46**	24.95
178	Basofil - p-aramid (Kevlar)	Soxtec	3	**0.92**	**0.14**	12.32

3.3.2 Agreed allowance

A number of experiments were performed on samples **136 (=179)**, **137** and **171** of pure melamine, in order to evaluate the agreed allowance of the new fibre. Samples **136 (=179)** and **171** were staple fibres and samples **137** and **180** were needed-

punched felts. The agreed allowance was calculated both for untreated and pre-treated samples.

The procedure described in the following, which takes into account terms and definition of UNI 9213 [4] and UNI 8048 [5], was applied. Weighing bottles were dried for 1 h in a ventilated oven at 105 ± 3 °C, then cooled in a dessicator and weighed. One sample of about 2 g of melamine was placed in each weighing bottle and dried for 4 hours in a ventilated oven at 105 ± 3 °C, then cooled in a dessicator and weighed. Samples were then conditioned for 72 hours at 20 ± 2 °C and 65 ± 2 % relative humidity and weighed immediately after the conditioning period. The following formulas were used to calculate the agreed allowance.

Water mass = wet sample mass – dried sample mass

Agreed allowance = (water mass / dried sample mass) * 100

Ten specimens per each sample were analysed (Table 7). Results showed values lower than 7.00 in the case of untreated samples and higher than 7.00 for pre-treated samples, with an average value for melamine's agreed allowance of 6.79 %.

Table 7: Agreed allowance for melamine (4 hours drying step).

JRC code	drying time	replicates	agreed allowance %	uncertainty at 95 %	RSD
136 pre-treated	4 h	10	7.34	0.05	1.03
136 untreated	4 h	10	5.75	0.03	0.78
137 pre-treated	4 h	10	7.59	0.05	0.93
137 untreated	4 h	10	6.51	0.03	0.69
171 pre-treated	4 h	10	7.28	0.02	0.45
171 untreated	4 h	10	6.27	0.04	0.97
average			6.79	0.14	9.83

Following discussions with experts from Member States, it was decided to confirm previous results by applying a slightly modified procedure. The weighting bottles were dried in a ventilated oven at 105 ± 3 °C for 5 h (instead of 1 h) and the drying step was of 16 h in a ventilated oven at 105 ± 3 °C (instead of 4 h). Results obtained with this procedure were in line with the ones evaluated with a drying step of 4 hours (Table 8). In addition, the range of values determined for untreated and pre-treated samples narrowed, with the average agreed allowance being 6.88 %.

Experts from Member States agreed to establish a value of 7.00 as agreed allowance for melamine.

The detailed results are reported in Annex V.

Table 8: Agreed allowance for melamine (16 hours drying step).

JRC code	drying time	replicates	agreed allowance %	uncertainty at 95 %	RSD
136/179 pre-treated	16 h	10	6.87	0.21	4.34
136/179 untreated	16 h	10	6.52	0.03	0.61
137 pre-treated	16 h	10	7.21	0.04	0.73
137 untreated	16 h	10	6.92	0.05	1.11
average			6.88	0.08	4.22

3.3.3 Chemical methods

Pre-treated specimens of about 1 g were analysed by chemical dissolution methods. Samples of pure melamine were tested with all the chemical methods described in Directive 96/73/EC [2]. In particular, method 1 (acetone), 2 (hypochlorite), 3 (formic acid and zinc chloride), 4 (formic acid, 80 w/w), 5 (benzyl alcohol), 6 (dichloromethane), 7 (sulphuric acid, 75% w/w), 8 (dimethylformamide), 9 (carbon disulphide/acetone, 55.5/44.5 v/v), 10 (glacial acetic acid), 11 (sulphuric acid, 75 w/w), 13 (xylene), 14 (concentrated sulphuric acid) and 15 (cyclohexanone) were applied. It was evidenced that melamine is insoluble in all the reagents traditionally used in the already validated quantitative methods. For this reason correction factors *d* for mass loss of the new fibre (insoluble component) in the reagents during analysis were evaluated.

The correction factors *d* were calculated using the following formula:

$$d = \frac{m}{r} \qquad 3.3.1$$

where:

m is the dry mass of the specimen after pre-treatment

r is the dry mass of the residue

All weighing operations were performed using an analytical balance with an uncertainty value of ± 0.0001 g.

In the case of compositional analysis of binary and ternary mixtures, after weighing, the residues were analysed by microscopy to verify the complete dissolution of the soluble component.

The percentages of insoluble component on a clean, dry mass basis, disregarding loss of fibre mass during pre-treatment, were calculated using the following formula:

$$P_1\ \% \ = \ \frac{100\ r\ d}{m} \qquad 3.3.2$$

where:

P_1 is the percentage of clean, dry insoluble component

m is the dry mass of the specimen after pre-treatment

r is the dry mass of the residue

d is the correction factor for loss of mass of the insoluble component in the reagent during analysis

Method 3 (formic acid and zinc chloride) and method 7 (sulphuric acid, 75% w/w) were applied in the case of mixtures melamine/cotton, viscose and Visil (a type of viscose with embedded silica), method 8 (dimethylformamide) for mixtures with modacrylic and method 14 (concentrated sulphuric acid) with mixtures with polyester or aramid (after verification that aramid is almost completely soluble in concentrated sulphuric acid).

In the case of binary mixtures, calculations of percentage of insoluble component on clean, dry mass basis, with adjustment by conventional factors (*agreed allowances*) and, where appropriate, correction factors d for loss of mass during pre-treatment, were performed using the following formula:

$$P_{1A}\ \% \ = \ \frac{100\ P_1\left(1+\frac{a_1+b_1}{100}\right)}{P_1\left(1+\frac{a_1+b_1}{100}\right)+\left(100-P_1\right)\left(1+\frac{a_2+b_2}{100}\right)} \qquad 3.3.3$$

where:

P_{1A} is the percentage of insoluble component, adjusted by *agreed allowances* and for loss of mass during pre-treatment

P_1 is the percentage of clean, dry insoluble component as calculated from equation 3.3.2

a_1 is the *agreed allowance* for the insoluble component (listed in Annex II to the Directive on textile names [1])

a_2 is the *agreed allowance* for the soluble component (listed in Annex II to the Directive on textile names [1])

b_1 is the percentage loss of insoluble component caused by the pre-treatment

b_2 is the percentage loss of soluble component caused by the pre-treatment

The percentage of the soluble component (P_{2A} %) was obtained by difference.

Correction factors b_1 and b_2 were ignored, as if the normal pre-treatment by extraction with light petroleum ether and water was applied. The *agreed allowances* used in the calculations are reported in Table 9. According to JRC experiments, for melamine the value of the *agreed allowance* used was 7.00. For Visil the value of the *agreed allowance* of viscose (13.00) was used in the calculations.

Table 9: *Agreed allowances* used in the calculations.

	agreed allowance
aramid	8.00
cotton	8.50
modacrylic	2.00
lyocell	13.00
polyester	1.50
viscose	13.00

3.3.4 Methods proposed by the applicant

The applicant proposed three new methods for the quantification of melamine in mixtures.

The first method makes use of 6.15 % sodium hypochlorite solution at 90°C with contact time of three hours. It was suggested to quantify melamine and, in principle, is applicable to binary fibre blends containing cellulosic fibres (such as cotton, viscose, Visil, lyocell), which are soluble in the reagent. This method, in combination with the second one, was also proposed in the analysis of mixtures of melamine with cellulosic fibres, along with modacrylic, polyester and other fibres which are not soluble in the reagent.

The second method is based on 50 % m/m sulphuric acid solution at 90 °C for three hours. It was recommended, in conjunction with the first method, to determine the melamine content of blends containing cellulosic fibres along with other fibres which are not soluble in the reagent.

The third method was proposed to quantify melamine when blended with aramid fibres using 90% m/m formic acid solution as reagent (three hours contact time at 90 °C). The descriptions of these methods are reported in Annex I.

3.3.5 Results

For each sample the homogeneity was verified and five to twenty replicate specimens were usually analysed. The petitioner indicated samples composition approximately and, for this reason, they should not be regarded as true reference values.

Table 10: Statistical tests used to confirm the presence of outliers.

	upper outlier	lower outlier	comments
"t-like" tests statistics	$T1 = \frac{x_n - \bar{x}}{s_x}$	$T1 = \frac{\bar{x} - x_1}{s_x}$	Test for single outlier, sometimes called T_n test
	$T2 = \frac{\sum (x_i - \bar{x})}{s_x}$	$T2 = \frac{\sum (\bar{x} - x_i)}{s_x}$	Block test for k upper or lower outliers
	$T3 = \frac{x_n - x_1}{s_x}$		Block test for one upper and one lower outlier
"Dixon-like" statistics	$T4 = \frac{x_n - x_{n-1}}{x_n - x_1}$	$T4 = \frac{x_2 - x_1}{x_n - x_1}$	Tests for a single outlier, sometimes called the Q test
	$T5 = \frac{x_n - x_{n-2}}{x_n - x_2}$	$T5 = \frac{x_3 - x_1}{x_{n-1} - x_1}$	Tests for a single outlier, a form of the Q-test that provides some protection from masking

The data were collected and subjected to statistical evaluation. The procedure followed guidelines ISO 5725 [6] and IUPAC harmonised protocol (1995) [7]. The results were examined for evidence of individual systematic error using statistical tests, as laid down in ISO 5725, in order to determine the presence of outliers. Only few outliers were found out of many hundreds of measurements and they were eliminated after confirmation using some other statistical tests [8], summarised in Table 10.

The valid results were then subjected to statistical evaluation. The average and standard deviation (SD) of each set of data were calculated, as well as the relative standard deviation (RSD). The RSD was used to measure the dispersion of the distribution of test results in one laboratory: the lower the value of RSD, the better the repeatability of the method. The confidence intervals (uncertainties) were calculated at 95 % of probability, using the following formula:

$$\mu = x_m \pm \frac{t\ s}{\sqrt{N}} \qquad 3.3.4$$

where:

t is the value listed in the Student's t-distribution for a certain number of degrees of freedom and level of probability

s is the estimated standard deviation

μ is the true value

x_m is the average of experimental results

N is the number of measurements

Annex V reports all the results regarding the evaluation of correction factors *d* and the composition analyses. All the measurements were performed at the JRC laboratories. An overview of the relevant results, with uncertainties calculated for a confidence level of 95 %, is shown in Tables 11-31. The number of replicates is reported either in a different column or in parenthesis.

The correction factors *d* for melamine were evaluated according to the already validated methods of the Directive 96/73/EC. Twenty specimens were analysed using each method, 10 replicates of sample **136** (staple fibre) and 10 of sample **137** (felt) to derive correction factors that are valid for different types of samples. Different linear densities were considered as melamine is produced only as staple fibre with a distribution of elliptical cross-section with different dimensions. The estimated values for *d* were generally equal to 1.01, except in the case of method 3 (formic acid and zinc chloride) and 10 (glacial acetic acid), for which *d* value was 1.00.

Table 11: Correction factors *d* for melamine, according to the methods of Directive 96/73/EC.

JRC code	method	replicates	melamine %	d	uncertainty at 95 %	RSD %
136 - 137	1	20	99.46	**1.006**	**0.001**	0.15
136 - 137	2	20	99.34	**1.007**	**0.001**	0.17
136 - 137	3	20	99.96	**1.000**	**0.001**	0.19
136 - 137	4	20	99.18	**1.009**	**0.001**	0.20
136 - 137	5	20	99.54	**1.005**	**0.001**	0.14
136 - 137	6	20	99.55	**1.005**	**0.001**	0.16
136 - 137	7	20	99.16	**1.009**	**0.001**	0.18
136 - 137	8	20	99.43	**1.006**	**0.001**	0.14
136 - 137	9	20	99.19	**1.009**	**0.001**	0.24
136 - 137	10	20	99.87	**1.001**	**0.001**	0.15
136 - 137	11	20	99.53	**1.005**	**0.002**	0.48
136 - 137	13	20	99.28	**1.008**	**0.001**	0.17
136 - 137	14	20	99.32	**1.007**	**0.001**	0.18
136 - 137	15	20	99.06	**1.010**	**0.001**	0.23

The correction factors *d* of melamine for methods 3, 4, 7, 8 and 14 were also evaluated through a ring trial, as described in a EUR report [9]. The interlaboratory trial results generally confirmed the values estimated at the JRC. A slight difference was found in the case of method 3, for which the *d* value was established equal to 1.01, and of the *d* value for method 7, which during the ring trial was obtained equal to 1.07. However, the repeatability and reproducibility limits calculated were very poor and the data could not be considered as being part of a normal distribution. One explanation of such poor results could be the very strong influence of temperature on the solubility properties of melamine with this method. In fact, results shown in Table 12 suggest that this parameter should be maintained strictly in the range 50 ± 2 °C to avoid a partial solubilisation of melamine. If temperature is raised to 55 °C almost 8 % of melamine is dissolved and this percentage attains 35 at 60 °C. The participants to the ring trial were advised of this critical point; however, method 7 states that the temperature should be maintained in the range 50 ± 5 °C, so perhaps JRC advice was not always followed.

For all these reasons, in agreement with national experts it was decided that method 7 is not applicable to blends containing melamine.

Table 12: Influence of temperature on pure melamine applying Method 7 of Directive 93/76/EC.

JRC code	temperature ° C	replicates	fibre %	uncertainty at 95 %	RSD %	d factor	uncertainty at 95 %	RSD %
136	48	5	**99.25**	**0.10**	0.10	**1.008**	**0.001**	0.104
136-137	50	10	**99.14**	**0.15**	0.21	**1.009**	**0.002**	0.227
136	52	5	**99.02**	**0.19**	0.19	**1.011**	**0.002**	0.202
136	55	5	**92.30**	**0.13**	0.14	**1.089**	**0.002**	0.152
136	60	5	**74.99**	**0.42**	0.55	**1.357**	**0.008**	0.577
136	70	5	**0.33**	**0.15**	0.15		**soluble**	

The three methods proposed by the applicant (see 3.3.4) were tested on pure melamine and some other fibres and subsequently *d* values were calculated. For all the proposed methods, contact time was three hours; the JRC studied the influence of this parameter performing analyses on pure melamine and fibre blends using three and one hour as contact time.

Table 13: Solubility properties of pure fibres (method with hot sulphuric acid).

JRC code	composition %	contact time	replicates	fibre %	unc. at 95 %	RSD %	d	unc. at 95 %	RSD %
136	100 % melamine	1 h	10	**99.54**	**0.13**	0.18			
136 - 137	100 % melamine	3 h	10	**99.97**	**0.01**	0.01			
137	100 % melamine	30 min	5	**99.97**	**0.03**	0.02			
137	100 % melamine	2.5 h	5	**99.95**	**0.03**	0.03			
171	100 % melamine	1 h	5	**99.95**	**0.18**	0.15			
153	100 % polyester	1 h	10	99.92	0.08	0.11	**1.001**	**0.001**	0.11
154	100 % modacrylic	1 h	10	99.76	0.06	0.09	**1.002**	**0.001**	0.09
150	100 % aramid (Kevlar)	1 h	10	99.32	0.04	0.05	**1.007**	**0.0004**	0.05
151	100 % aramid (Nomex)	1 h	10	99.35	0.05	0.07	**1.007**	**0.001**	0.07
152	100 % lyocell (Tencel)	1 h	10	**99.25**	**0.04**	0.05			
034	100 % viscose	1 h	5	**99.49**	**0.03**	0.03			
038	100 % polyamide	1 h	5	**99.77**	**0.13**	0.10			
159-005	100 % cotton	1 h	5	**99.30**	**0.03**	0.03			
040	100 % triacetate	1 h	5	**99.81**	**0.02**	0.02			

As shown by results, melamine is soluble both in 50 % sulphuric acid and 90 % formic acid solutions at 90 °C. In both cases, one hour is sufficient to dissolve melamine.

Apart from melamine, also polyamide, triacetate and cellulosic fibres (such as cotton, viscose and lyocell) are soluble with the hot sulphuric acid method, whereas polyester, modacrylic, meta and para aramid are insoluble.

In the case of the hot formic acid method, among the tested fibres only polyamide and melamine are soluble; cotton, meta and para aramid are insoluble, whereas viscose, lyocell, polyester and modacrylic are partially soluble.

Table 14: Solubility properties of pure fibres (method with hot formic acid).

JRC code	composition %	contact time	replicates	fibre %	unc. at 95 %	RSD %	d	unc. at 95 %	RSD %
136 - 137	100 % melamine	1 h	10	**99.94**	**0.01**	0.02			
136 - 137	100 % melamine	3 h	10	**99.96**	**0.02**	0.02			
171	100 % melamine	1 h	5	**99.92**	**0.10**	0.08			
057 - 150	100 % aramid (Kevlar)	1 h	10	98.72	0.08	0.11	**1.014**	**0.001**	0.12
151	100 % aramid (Nomex)	1 h	8	98.77	0.19	0.24	**1.013**	**0.002**	0.27
038	100 % polyamide	1 h	5	**99.86**	**0.14**	0.12			
159	100 % cotton	1 h	5	99.65	0.08	0.06	**1.004**	**0.001**	0.07
034	100 % viscose	1 h	5	63.97	0.11	0.14	**1.636**	**0.003**	0.15
152	100 % lyocell (Tencel)	1 h	5	95.11	0.29	0.24	**1.058**	**0.004**	0.27
153	100 % polyester	1 h	5	90.65	0.16	0.15	**1.105**	**0.002**	0.15
154	100 % modacrylic	1 h	7	81.63	0.76	1.01	**1.230**	**0.012**	1.02

The method with hot 6.15 % NaClO solution dissolves cotton, viscose and lyocell within one hour contact time, whereas polyester and modacrylic are insoluble. However, in contrast with the petitioner's statements, this method is not applicable to

melamine because of its partial solubility in this reagent. Due to these discrepancies in results, the JRC requested further information and the petitioner responded that the reagent used in the company for this method was a commercial product available in the USA for surface cleaning. This product is not available in Europe and the applicant did not provide it to the JRC. Some experiments were performed using a home prepared reagent, based on the formulation of the commercial product used by Basofil, however results confirmed the partial solubility of melamine. In any case, commercial products cannot be used for analytical purposes as they are not analytical grade reagents and their formulation can vary over time.

As a further attempt, method 2 of Directive 96/73/EC, which makes use of a sodium hypochloride solution with a slightly different concentration, was applied to pure melamine and lyocell. The experimental conditions of method 2 were changed; the temperature was set at 90 °C and the contact time at one or three hours, to be comparable with the conditions of the method proposed by the applicant. Even in this case, however, results confirmed the partial solubility of melamine which increases up to 30 % for a contact time of three hours, whereas lyocell is completely soluble within one hour.

Table 15: Solubility properties of pure fibres (method with hot sodium hypochloride solution and method 2 at 90 °C).

JRC code	composition %	contact time	replicates	fibre %	unc. at 95 %	RSD %	d	unc. at 95 %	RSD %
136-137	100 % melamine	3 h	10	93.55	0.26	0.40	**1.074**	**0.003**	0.43
136	100 % melamine	1 h	11	94.29	0.27	0.42	**1.065**	**0.003**	0.45
005	100 % cotton	30 min	5	**99.94**	**0.02**	0.02			
005	100 % cotton	1 h	5	**99.96**	**0.02**	0.02			
034	100 % viscose	1 h	10	**99.94**	**0.01**	0.02			
152	100 % lyocell (Tencel)	1 h	10	**99.95**	**0.01**	0.02			
153	100 % low melt polyester	1 h	10	97.38	0.06	0.08	**1.027**	**0.001**	0.08
154	100 % modacrylic	1 h	10	98.85	0.07	0.10	**1.012**	**0.001**	0.10
M2-136	100 % melamine	1 h	5	95.08	0.12	0.10	**1.055**	**0.001**	0.11
M2-136	100 % melamine	3 h	5	69.11	0.87	1.02	**1.478**	**0.019**	1.06
M2-152	100 % lyocell (Tencel)	1 h	5	**99.78**	**0.12**	0.10			

Due to the fact that the composition of blends provided by the producer is only indicative and that manual separation of samples was not possible for the type of structure (each yarn is a blend), there were no reference values for the composition of samples to be compared with results obtained with the methods proposed by the applicant, in order to evaluate their accuracy. For this reason, some tests were carried out on binary mixtures melamine/aramid with method 14 (after verifying that aramid

is almost completely soluble in concentrated sulphuric acid) to be compared with the quantification done with the hot formic acid method which dissolves melamine. Unfortunately, method 14 seemed to be inadequate. Several problems were experienced due to the fact that the reagent (pure sulphuric acid) shows a high density and probably had difficulties entering the sample structure and dissolving aramid. To improve the situation, samples were manually dissected and in some cases frozen with liquid nitrogen and immediately milled as much as possible to increase the surface of samples. Despite the accurate sample preparation, even if results were better compared to the first ones, in particular using liquid nitrogen in the preparation, they could not be trusted as reference values. Moreover, in certain cases they were very different both from the stated composition and the results obtained with the hot formic acid method (see also Tab. 18).

Table 16: Analysis of composition performed by method 14 of Directive 96/73/EC in mixtures melamine/aramid

JRC code	composition %	method	sample preparation	replicates	MLF %	unc. at 95 %	RSD %
138	40 MLF - 60 Nomex	14		10	**46.08**	**0.52**	1.57
			liquid nitrogen	10	**39.22**	**0.33**	1.19
139	40 MLF - 60 Nomex	14	liquid nitrogen	10	**30.49**	**2.38**	10.90
140	50 MLF - 50 Nomex 462	14		10	**51.33**	**0.53**	1.44
142	40 MLF - 60 Kevlar	14		10	**83.13**	**0.37**	0.63
			liquid nitrogen	10	**73.15**	**0.52**	0.99
143	25 MLF - 75 Conex	14	liquid nitrogen	8	**18.82**	**1.08**	6.86

All the methods proposed by the applicants foresaw a contact time of three hours. The JRC evaluated the influence of this parameter and performed analyses on pure melamine and binary blends with aramid using one and three hours contact time (see Tables 17-19). The average contents of melamine, obtained in these experimental conditions, were statistically compared. Before this, the precision (standard deviations) of the two independent set of measurements were compared in order to judge if variances could be considered homogeneous or not and thus to know which formulas had to be used to compare means [8].

To compare the precision, the ratio of samples variances was calculated:

$$F\ value = s^2_{x/}\, s^2_y \qquad 3.3.5$$

where:

s^2_x variance of measurement set x

s^2_y variance of measurement set y

The *F value* must be larger than one, so that the larger variance is s^2_x. *F values* were compared with *F critical* values reported in tables that take into account the degrees of freedom in the measurement sets x and y and the confidence level required. If *F value* > *F critic* that the two sets of measurements do not have homogeneous precision.

To compare the means of two independent sets of measurements we used the standardised difference of sample means (see Eq. 3.3.6).

$$T = \frac{d}{s_d} \qquad 3.3.6$$

where:

d difference between sample means of the two groups

s_d standard deviation of the difference of means

T value was compared with *T critical* listed in the Student's t-distribution tables (at a certain level of probability and degrees of freedom). If *T value* is in the range ± *T critical*, then the two averages can be considered equivalent.

Two cases have to be distinguished: the first one assumes that the two sets have the same precision and the second one that the two sets of data have different precisions.

In the case of comparable precisions, the standard deviation, s_d, of the difference of means is calculated as in the following:

$$s_d = s_{pool} * \sqrt{\frac{1}{n_x} + \frac{1}{n_y}} \qquad 3.3.7$$

with

$$s_{pool} = \sqrt{\frac{\nu_x * s_x^2 + \nu_y * s_y^2}{\nu_x + \nu_y}} \qquad 3.3.8$$

and

$$\nu = n_x + n_y - 2 \qquad 3.3.9$$

where:

n_x number of replicates for measurement set x

n_y number of replicates for measurement set y

v_x degrees of freedom of measurement set x

v_y degrees of freedom of measurement set y

v degrees of freedom

If the two groups of measurements do not share the same variance, then it is not possible to pool the data and the following formulas apply:

$$s_d = \sqrt{\frac{s_x^2}{n_x} + \frac{s_y^2}{n_y}} \qquad 3.3.10$$

$$v = \frac{v_x * v_y}{v_y * c^2 + v_x * (1-c)^2} \qquad 3.3.11$$

$$c = \frac{1}{n_x} * \left(\frac{s_x^2}{s_d^2} \right) \qquad 3.3.12$$

If necessary, the calculated value of v should be rounded down to the nearest integer. The level of probability chosen was 95 %.

Table 17: Influence of contact time in the hot sulphuric acid method.

JRC code	composition %	contact time	replicates	MLF %	unc. at 95 %	RSD %
136-137-171	100 MLF	1 h	5	**99.95**	**0.18**	0.15
		3 h	10	**99.97**	**0.01**	0.01
138	40 MLF - 60 aramid (Nomex)	1 h	10	**36.06**	**0.29**	1.12
		3 h	10	**35.89**	**0.24**	0.95
139	40 MLF - 60 aramid (Nomex)	1 h	10	**32.89**	**0.25**	1.05
		3 h	10	**29.36**	**0.28**	1.31
140	50 MLF - 50 aramid (Nomex 462)	1 h	10	**47.73**	**0.25**	0.73
		3 h	10	**48.20**	**0.18**	0.51
141	40 MLF - 60 aramid (Kevlar)	1 h	10	**37.55**	**0.48**	1.79
		3 h	10	**38.53**	**0.13**	0.46
142	40 MLF - 60 aramid (Kevlar)	1 h	10	**35.55**	**0.36**	1.42
		3 h	10	**35.64**	**0.19**	0.73
143	25 MLF - 75 aramid (Conex)	1 h	10	**15.33**	**0.20**	1.78
		3 h	10	**15.08**	**0.29**	2.72
177	MLF - aramid (Nomex)	1 h	10	**32.27**	**0.20**	0.88
178	MLF - aramid (Kevlar)	1 h	10	**46.12**	**0.06**	0.19

Table 18: Influence of contact time in the hot formic acid method.

JRC code	composition %	contact time	replicates	MLF %	unc. at 95 %	RSD %
136-137	100 MLF	1 h	10	**99.94**	**0.01**	0.02
		3 h	10	**99.96**	**0.02**	0.02
138	40 MLF - 60 aramid (Nomex)	1 h	10	**39.67**	**0.31**	1.10
		3 h	10	**39.66**	**0.31**	1.10
139	40 MLF - 60 aramid (Nomex)	1 h	20	**34.66**	**0.28**	1.45
		3 h	20	**35.22**	**0.54**	3.30
140	50 MLF - 50 aramid (Nomex 462)	1 h	20	**48.52**	**0.42**	1.87
		3 h	10	**48.64**	**0.15**	0.45
141	40 MLF - 60 aramid (Kevlar)	1 h	10	**38.46**	**0.15**	0.55
		3 h	10	**38.32**	**0.08**	0.29
142	40 MLF - 60 aramid (Kevlar)	1 h	10	**39.84**	**0.16**	0.56
		3 h	10	**39.71**	**0.11**	0.40
143	25 MLF - 75 aramid (Conex)	1 h	10	**18.61**	**0.17**	1.28
		3 h	10	**18.70**	**0.29**	2.16
177	MLF - aramid (Nomex)	1 h	10	**34.94**	**0.17**	0.68
178	MLF - aramid (Kevlar)	1 h	15	**46.43**	**0.13**	0.51

As shown in Table 19, the methods based on hot sulphuric and formic acids gave the same results on pure melamine applying three or one hour as contact time. Viceversa, statistically different results were obtained in the case of the hot sodium hypochloride method, where melamine was dissolved more, increasing the contact time from one to three hours.

Results on quantification of blends melamine/aramid did not evidence statistically significant differences in the case of the hot formic acid method among one and three hours contact time. On the contrary, in the case of the hot sulphuric acid method, out

of six binary mixtures analysed, differences were noticed for samples **139**, **140** and **141**.

Table 19: Influence of contact times in the methods proposed by the applicant (one vs three hours).

method	JRC code	F	F crit	SD	T	T crit	average
50 % H2SO4	171-136-137	166.39	3.63	≠	0.3060	2.7765	=
	138	1.42	3.18	=	1.0403	2.1009	=
	139	1.24	3.18	=	21.5831	2.1009	≠
	140	1.99	3.18	=	-3.4302	2.1009	≠
	141	14.13	3.18	≠	-4.4552	2.2281	≠
	142	3.69	3.18	≠	-0.5098	2.1009	=
	143	2.25	3.18	=	1.5883	2.1009	=
90 % HCOOH	136-137	1.29	3.18	=	1.9652	2.1009	=
	138	1.00	3.18	=	0.0422	2.1009	=
	139	5.35	2.17	≠	-1.9713	2.0595	=
	140	17.53	2.95	≠	-0.5808	2.0687	=
	141	3.47	3.18	≠	1.7837	2.1448	=
	142	1.95	3.18	=	1.5121	2.1009	=
	143	2.85	3.18	=	-0.6216	2.1009	=
6.15 % NaOCl	136-137	1.09	3.02	=	-4.3837	2.093	≠

Tables 20 and 21 show the same results presented in Tables 17 and 18, ordered in a different way to facilitate comparison among quantitative results obtained with the two hot acid methods. Averages were compared statistically to verify if the two methods provide results that can be considered equivalent. Both methods are supposed to dissolve melamine; however the hot sulphuric acid method always provided lower percentages of melamine in the analysed mixtures and these differences, as shown in Table 22, can be considered statistically significant. The hot sulphuric acid method seemed not to be able to completely dissolve melamine when in mixture with aramid, both using one and three hours contact time. These conclusions were also supported by the microscopic analysis of residues which still contained some left melamine. Furthermore, even if the compositions indicated by the producer are quite rough and can not be considered as reference values, the values obtained with formic acid method were closer to these stated compositions. In particular, this is true considering that a manufacturing tolerance of 3 % shall be permitted between the stated fibre percentages and the composition obtained from analysis, in relation to the total weight of fibres shown on the label (as foreseen by article 6, comma 4b in Directive 96/74/EC on textile names). Only sample **139** and **143** are exceptions and this is probably most probably due to wrong labelling.

Table 20: Comparison between hot formic and sulphuric acid methods (contact time of 1 hour).

JRC code	composition %	method	replicates	MLF %	unc. at 95 %	RSD %
136-137	100 MLF	90% HCOOH	10	**99.94**	**0.01**	0.02
		50% H2SO4	5	**99.54**	**0.13**	0.18
138	40 MLF - 60 aramid (Nomex)	90% HCOOH	10	**39.67**	**0.31**	1.10
		50% H2SO4	10	**36.06**	**0.29**	1.12
139	40 MLF - 60 aramid (Nomex)	90% HCOOH	20	**34.66**	**0.28**	1.45
		50% H2SO4	10	**32.89**	**0.25**	1.05
140	50 MLF - 50 aramid (Nomex)	90% HCOOH	20	**48.52**	**0.42**	1.87
		50% H2SO4	10	**47.73**	**0.25**	0.73
141	40 MLF - 60 aramid (Kevlar)	90% HCOOH	10	**38.46**	**0.15**	0.55
		50% H2SO4	10	**37.55**	**0.48**	1.79
142	40 MLF - 60 aramid (Kevlar)	90% HCOOH	10	**39.84**	**0.16**	0.56
		50% H2SO4	10	**35.55**	**0.36**	1.42
143	25 MLF - 75 aramid (Conex)	90% HCOOH	10	**18.61**	**0.17**	1.28
		50% H2SO4	10	**15.33**	**0.20**	1.78
177	MLF - aramid (Nomex)	90% HCOOH	10	**34.94**	**0.17**	0.68
		50% H2SO4	10	**32.27**	**0.20**	0.88
178	MLF - aramid (Kevlar)	90% HCOOH	15	**46.43**	**0.13**	0.51
		50% H2SO4	10	**46.12**	**0.06**	0.19

Table 21: Comparison between hot formic and sulphuric acid methods (contact time of 3 hours).

JRC code	composition %	method	replicates	MLF %	unc. at 95 %	RSD %
136-137	100 MLF	90% HCOOH	10	**99.96**	**0.02**	0.02
		50% H2SO4	10	**99.97**	**0.01**	0.01
138	40 MLF - 60 aramid (Nomex)	90% HCOOH	10	**39.66**	**0.31**	1.10
		50% H2SO4	10	**35.89**	**0.24**	0.95
139	40 MLF - 60 aramid (Nomex)	90% HCOOH	20	**35.22**	**0.54**	3.30
		50% H2SO4	10	**29.36**	**0.28**	1.31
140	50 MLF - 50 aramid (Nomex)	90% HCOOH	10	**48.64**	**0.15**	0.45
		50% H2SO4	10	**48.20**	**0.18**	0.51
141	40 MLF - 60 aramid (Kevlar)	90% HCOOH	10	**38.32**	**0.08**	0.29
		50% H2SO4	10	**38.53**	**0.13**	0.46
142	40 MLF - 60 aramid (Kevlar)	90% HCOOH	10	**39.71**	**0.11**	0.40
		50% H2SO4	10	**35.64**	**0.19**	0.73
143	25 MLF - 75 aramid (Conex)	90% HCOOH	10	**18.70**	**0.29**	2.16
		50% H2SO4	10	**15.08**	**0.29**	2.72

Table 22: Comparison between hot formic and sulphuric acid methods.

contact time	JRC code	F	F crit	SD	T	T crit	average
1 h	**171**	3.36	6.39	=	-0.4197	2.3060	=
	136-137	88.23	3.18	≠	-6.8504	2.2622	≠
	138	1.16	3.18	=	19.1494	2.1009	≠
	139	2.11	2.95	=	9.9549	2.0484	≠
	140	6.77	2.95	≠	3.3931	2.1448	≠
	141	10.29	3.18	≠	4.0443	2.2010	≠
	142	5.17	3.18	≠	24.7065	2.1788	≠
	143	1.31	3.18	=	28.5581	2.1009	≠
	177	1.42	3.18	=	22.7280	2.1009	≠
	178	7.16	3.03	≠	4.5894	2.1604	≠
3 h	**136-137**	3.79	3.18	≠	0.7917	2.1604	=
	138	1.65	3.18	=	21.5462	2.1009	≠
	139	9.12	2.95	≠	20.4080	2.1604	≠
	140	1.30	3.18	=	4.2749	2.1009	≠
	141	2.53	3.18	=	-3.1977	2.1009	≠
	142	2.73	3.18	=	42.0903	2.1009	≠
	143	1.03	3.18	=	19.9021	2.1009	≠

All the three methods proposed by the petitioner foresaw the dissolution process at 90 °C. The JRC investigated the influence of temperature performing a set of analyses, on pure melamine, by varying this parameter. The contact time was set to one hour. For comparison purposes, some experiments were conducted on pure melamine also applying a modified version of method 2 of Directive 96/73/EC, the modification being a different temperature.

The statistical analysis, reported in Table 24, evidenced a strong influence of temperature on the solubility properties of melamine. In the case of the hot sodium hypochlorite method, the percentage of melamine dissolved increased together with the temperature, starting from around 4 % at 80 °C up to around 12 % at 95 °C. The same trend was also observed for method 2 in Directive 96/73/EC, applied at 70, 80 and 90 °C. In the case of the hot sulphuric acid method, results obtained at 80 and 85 °C cannot be considered equivalent, due to an incomplete solubilisation of melamine at 80 °C. On the contrary, results obtained a 85 °C are statistically comparable to the ones evaluated at 90 °C. Finally for the hot formic acid method, the statistics suggested that results obtained at 88, 90 and 92 °C can not be considered equal. However, this conclusion is probably due to the very low value of standard deviation and, considering the usual variability of results for replicates analysed with dissolution methods already validated, results should be considered comparable.

Table 23: Influence of temperature on methods proposed by the applicant and a modified version of method 2 of Directive 96/73/EC (contact time 1 h).

JRC code	method	T °C	replicates	MLF %	unc. at 95 %	RSD %	d	unc. at 95 %	RSD %
136	50% H2SO4	80	5	**98.52**	**0.42**	0.42			
136		85	5	**99.79**	**0.23**	0.19			
171		90	5	**99.95**	**0.18**	0.15			
136	90% HCOOH	88	5	**99.86**	**0.05**	0.04			
136 - 137		90	10	**99.94**	**0.01**	0.00			
136		92	5	**99.88**	**0.03**	0.02			
136	6.15% NaClO	80	5	**95.95**	**0.21**	0.17	**1.045**	**0.002**	0.18
136		85	5	**95.52**	**0.20**	0.17	**1.050**	**0.002**	0.18
136		90	11	**94.29**	**0.27**	0.42	**1.065**	**0.003**	0.45
136		95	10	**88.55**	**0.60**	1.00	**1.138**	**0.008**	1.08
136	M2 - NaClO	70	5	**97.64**	**0.13**	0.11	**1.026**	**0.001**	0.11
136		80	5	**96.90**	**0.08**	0.07	**1.034**	**0.001**	0.07
136		90	5	**95.08**	**0.12**	0.10	**1.055**	**0.001**	0.11

Table 24: Influence of temperature on methods proposed by the applicant and a modified version of method 2 of Directive 96/73/EC (contact time 1 h).

method	JRC code	T °C	F	F crit	SD	T	T crit	average
50 % H2SO4	**136**	80-85	3.18	6.39	=	-7.3365	2.3060	≠
	136-171	85-90	0.61	6.39	=	1.4555	2.3060	=
90 % HCOOH	**136-137**	88-90	4.10	3.63	≠	-4.7121	2.5706	≠
	136-138	90-92	1.52	3.63	=	-5.7289	2.1604	≠
6.15 % NaClO	**136**	80-85	1.10	6.39	=	4.2400	2.3060	≠
	136	85-90	6.39	5.96	≠	-8.8068	2.1448	≠
	136	90-95	4.96	3.02	≠	18.8224	2.1788	≠
M2 - NaClO	**136**	70-80	2.77	6.39	=	13.5327	2.3060	≠
	136	80-90	2.22	6.39	=	-35.9912	2.3060	≠

The influence of the temperature was investigated also in the case of binary blends with aramid (samples **138**, **141**, **142**) with the hot formic acid method. Again the statistical evaluation of results (Tab. 26) indicated that quantitative results strongly depended on this parameter which should be carefully controlled among 90 ± 2 °C In fact a larger range, such as 90 ± 5 °C does not provide comparable results.

Table 25: Influence of temperature on hot formic acid method (contact time 1 h).

JRC code	composition %	T °C	replicates	MLF %	unc. at 95 %	RSD %	other %	unc. at 95 %	RSD %
138	40 MLF - 60 aramid (Nomex)	80	10	**9.37**	**0.20**	2.92	**90.63**	**0.20**	0.30
		85	10	**19.36**	**0.19**	1.39	**80.64**	**0.19**	0.33
		90	10	**39.67**	**0.00**	1.10	**60.33**	**0.00**	0.72
		95	5	**38.86**	**0.35**	0.72	**61.14**	**0.35**	0.46
141	40 MLF - 60 aramid (Kevlar)	80	13	**6.28**	**0.49**	0.86	**93.72**	**0.49**	0.86
		85	6	**21.29**	**0.71**	3.17	**78.71**	**0.71**	0.86
		88	13	**38.30**	**0.25**	1.06	**61.70**	**0.25**	0.66
		90	10	**38.46**	**0.15**	0.55	**61.54**	**0.15**	0.34
		92	10	**38.68**	**0.21**	0.75	**61.32**	**0.21**	0.47
		95	10	**38.78**	**0.35**	1.25	**61.22**	**0.35**	0.79
142	40 MLF - 60 aramid (Kevlar)	80	10	**6.22**	**0.00**	10.42	**93.78**	**0.00**	0.69
		85	10	**12.58**	**0.27**	3.03	**87.42**	**0.27**	0.44
		90	10	**39.84**	**0.16**	0.56	**60.16**	**0.16**	0.37
		95	5	**39.48**	**0.29**	0.59	**60.52**	**0.21**	0.39

Table 26: Influence of temperature on hot formic acid method (contact time 1 h).

JRC code	T °C	F	F crit	SD	T	T crit	average
138	80-90	2.55	3.18	=	-185.9396	2.1009	≠
	85-90	2.64	3.18	=	-125.2292	2.1009	≠
	90-95	2.46	6.00	=	-3.7449	2.1009	≠
141	80-90	14.94	3.07	≠	-137.3531	2.1450	≠
	85-90	10.40	3.48	≠	-60.4626	2.2280	≠
	88-90	3.77	3.07	≠	-0.9998	2.1100	=
	90-92	1.92	3.18	≠	2.0191	2.1009	=
	90-95	5.36	3.18	≠	1.9294	2.1790	=
142	80-90	8.57	3.18	≠	-155.3368	2.2010	≠
	85-90	2.97	3.18	=	-195.6405	2.1010	≠
	90-95	1.12	3.63	=	-2.9307	2.1600	≠

The influence of the concentration of the formic acid solution was studied in the case of a binary mixture melamine-cotton (sample **175**). Results did not strongly depend on this parameter and the statistics reported in Table 28 shows that the concentration is not a critical parameter within the range 87 - 98-100% of formic acid.

Table 27: Influence of acid concentration on hot formic acid method (contact time 1 h).

JRC code	concentration %	replicates	MLF %	uncertainty at 95 %	RSD %	cotton %	uncertainty at 95 %	RSD %
175	98-100	3	**58.87**	**0.10**	0.07	**41.13**	**0.10**	0.10
175	93	3	**59.02**	**0.40**	0.28	**40.98**	**0.40**	0.40
175	90	5	**58.78**	**0.18**	0.25	**41.22**	**0.18**	0.35
175	87	3	**58.58**	**0.19**	0.13	**41.42**	**0.19**	0.19
175	85	5	**57.02**	**0.15**	0.25	**42.98**	**0.15**	0.34
175	80	5	**56.45**	**0.08**	0.14	**43.55**	**0.08**	0.18

Table 28: Influence of acid concentration on hot formic acid method (contact time 1 h).

JRC code	concentration %	F	F crit	SD	T	T crit	average
	80-90	3.54	6.39	=	-31.8723	2.3060	≠
	85-90	1.01	6.39	=	-19.2124	2.3060	≠
175	87-90	3.52	19.20	=	-2.2006	2.4470	=
	90-93	1.27	6.94	=	2.1696	2.4470	=
	90-98	13.16	19.20	=	0.9652	2.4470	=

Binary mixtures were quantified using the proper methods mentioned on the Directive, or the method proposed by the applicant. As the company did not specify the per cent composition of the blends, for comparison purposes several methods were used for the quantification. Before applying chemical dissolution methods, samples were dissected as much as possible in order to separate each individual yarn to let the reagent enter the structure more easily. Generally the agreement among results obtained with different methods was very good, except in the case of the quantification of sample **173** with method 3, which gave a completely different percentage of melamine from the ones calculated with data of methods 7 and 14. One possible explication is that this blend contains Visil, which is not a normal viscose but a modified viscose with embedded silica that perhaps cannot be dissolved with the reagent of method 3. As already mentioned above, quantification obtained with the hot sulphuric acid is not comparable with results obtained with the hot formic acid method. Because of data on solubility of cotton reported in Table 14, the field of application of the hot formic acid method could be enlarged to include the quantification of blends melamine/cotton (see sample **175**). The *d* correction factor for cotton was confirmed during the ring trial.

In the case of ternary and quaternary mixtures the quantification should be performed using at least two of the four possible variants, described in the Directive 73/44/EEC [10] and summarised in the following for a ternary mixture.

Variant 1 makes use of two different test specimens. One component (a) is dissolved from the first specimen and another component (b) is dissolved from the second one. Variant 2 foresees the use of two different test specimens. One component (a) is dissolved from the first test specimen and two components (a and b) from the second test specimen. For variant 3 two different test specimens are analysed. Two components (a and b) are dissolved from the first one and two components (b and c) from the second one. Variant 4 uses only one test specimen. One fibre is dissolved at a time on the same specimen/residue.

Table 29: Analysis of composition performed by different chemical methods.

JRC code	stated composition	method	T °C	contact time	replicates	MLF %	unc. at 95 %	RSD %
172	Melamine - Viscose	M3 ZnCl2, 85%HCOOH	40	2.5 h	10	**49.67**	**0.32**	0.91
		M7-50%H2SO4	50	1 h	15	**49.20**	**0.30**	1.11
		M14 -H2SO4	RT	10 +10 min	10	**49.76**	**0.44**	1.24
173	Melamine - Visil	M3 ZnCl2, 85%HCOOH	40	2.5 h	10	**85.02**	**0.15**	0.25
		M7-50%H2SO4	50	1 h	10	**20.21**	**0.13**	0.87
		M14 -H2SO4	RT	10 +10 min	10	**20.09**	**0.12**	0.83
174	Melamine - PES	M7-50%H2SO4	50	1 h	10	**39.51**	**0.14**	0.49
		M14 -H2SO4	RT	10 +10 min	10	**39.83**	**0.22**	0.78
175	Melamine - Cotton	90% HCOOH	90	1 h	10	**60.32**	**0.17**	0.40
		M7-50%H2SO4	50	1 h	10	**60.92**	**0.40**	0.92
		M14 -H2SO4	RT	10 +10 min	10	**61.23**	**0.33**	0.75
176	Melamine - Viscose	M3 ZnCl2, 85%HCOOH	40	2.5 h	10	**41.44**	**0.14**	0.53
		M7-50%H2SO4	50	1 h	10	**40.67**	**0.12**	0.40
		M14 -H2SO4	RT	10 +10 min	10	**40.72**	**0.23**	0.77
177	Melamine - m-aramid (Nomex)	90% HCOOH	90	1 h	10	**34.94**	**0.17**	0.68
		50% H2SO4	90	1 h	10	**32.27**	**0.20**	0.88
178	Melamine - p-aramid (Kevlar)	90% HCOOH	90	1 h	15	**46.43**	**0.13**	0.51
		50% H2SO4	90	1 h	10	**46.12**	**0.06**	0.19

Table 30: Analysis of composition performed by chemical methods.

JRC code	composition %	method	contact time	variant
144	17 MLF - 17 modacrylic - 66 PES	method 8 (DMF) 50% H2SO4, 90 °C	1 h + 30 min 1 h	4
144	17 MLF - 17 modacrylic - 66 PES	method 8 (DMF) 50% H2SO4, 90 °C	1 h + 30 min 3 h	4
144	17 MLF - 17 modacrylic - 66 PES	method 8 (DMF) method 14 (H2SO4)	1 h + 30 min 10 min + 10 min	2
145	12 MLF - 34 modacrylic - 24 PES - 30 lyocell	method 7 (H2SO4 75%) method 8 (DMF) method 14 (H2SO4)	1 h 1 h + 30 min 10 min + 10 min	4
145	12 MLF - 34 modacrylic - 24 PES - 30 lyocell	method 14 (H2SO4) method 7 (H2SO4 75%) method 8 (DMF)	10 min + 10 min 1 h 1 h + 30 min	2

JRC code	variant	replicates	MLF %	unc. at 95 %	modacrylic %	unc. at 95 %	PES %	unc. at 95 %	lyocell %
144	**4**	10	**17.09**	0.16	**16.00**	**0.36**	**66.99**	**0.34**	
144	**4**	5	**17.19**	0.15	**16.84**	**0.20**	**66.04**	**0.17**	
144	**2**	15	**18.26**	0.26	**16.27**	**0.16**	**65.47**	**0.30**	
145	**4**	10	**12.92**	0.39	**32.60**	**0.65**	**22.24**	**0.59**	**32.25**
145	**2**	10	**12.23**	0.05	**33.41**	**0.22**	**23.88**	**0.36**	**30.48**

Table 31: Evaluation of correction factors *d* for modacrylic and lyocell.

JRC code	composition %	method	contact time	replicates	fibre %	d	unc. at 95 %	RSD %
154	100 modacrylic	7	1 h	20	99.35	**1.007**	**0.0009**	0.19
152	100 lyocell	8	1 h + 30 min	20	99.85	**1.002**	**0.0004**	0.09

For samples **144** and **145** variant 2 and 4 were applied. In order to perform the quantification, the correction factor *d* for modacrylic with method 7 and for lyocell with method 8 had to be evaluated (as they are not available in the Directive). These correction factors are respectively 1.01 and 1.00 (see Tab. 31).

In the case of sample **144**, applying variant 4 modacrylic was dissolved first with method 8, then melamine with the hot sulphuric acid method either with one or three hours of contact time. Alternatively, applying variant 2, on one specimen modacrylic was dissolved with method 8 and on a second specimen modacrylic and polyester were dissolved using method 14. The results obtained with the two variants are similar even if not completely in agreement, probably due to the difficulty to the hot sulphuric acid method to dissolve completely the present melamine. Moreover they are in line with the stated composition of the sample.

Sample **145** was a quaternary blend. Also in this case variant 4 was carried out by dissolving the fibre components one by one: first of all lyocell with method 7, then modacrylic with method 8 and finally polyester with method 14. As a confirmation, variant 2 was performed solubilising: polyester, lyocell and modacrylic with method 14 on one specimen; lyocell with method 7 on a second specimen and modacrylic with method 8 on a third specimen. The quantification obtained by the two variants was quite comparable and in agreement with the stated composition.

Based on the presented experimental data, the following conclusions related to the three proposed methods could be drawn and agreed with experts from Member States.

The method which uses a 6.15 % sodium hypochloride solution at 90 °C was considered not adequate, mainly because it dissolves partially melamine, whereas it was not supposed to dissolve it. Also the 50 % sulphuric acid method at 90 °C was considered not adequate as it showed some difficulties in the complete dissolution of melamine, especially in the quantification of this fibre in mixtures.

On the contrary, the hot formic acid method was judged adequate for the analysis of mixtures containing melamine/aramid or melamine/cotton. As one and three hours contact time resulted in equivalent quantification both on pure melamine and on binary mixture samples, which represent products expected to be found on the market, experts from Member States considered that it would have been better to validate the method in a ring trial using one hour as contact time, so that the method would be less time-consuming and consequently less expensive.

The JRC then organised a ring trial with the participation of seventeen European laboratories and validated the hot formic acid method for binary blends melamine/aramid and melamine/cotton, as described in a EUR report [9]. The validation exercise was organised in accordance with the rules laid down in ISO 5725 (1994) [6]. Two binary blends with aramid and one with cotton were analysed, moreover two samples of pure aramid (meta and para) and one of cotton were tested in order to establish their correction factor *d*.

The validation was successful and the minimum number of valid results (8) was attained for every sample. The precision of the test method should be quoted, as a percentage by mass. The calculated repeatability standard deviation was 0.27 and the reproducibility standard deviation 0.58. The correction factors *d* for aramid and cotton were evaluated in the same context and the established values are 1.02 for both fibres.

4. Conclusions

The tests performed at the JRC described in this report confirmed that test methods are available for the identification, quantification and characterisation of the new fibre melamine.

Regarding the identification methods, the experimental results conducted at JRC showed that microscopic analysis is not a unique technique for the identification of this fibre and can lead to mistakes in the identification of man-made fibres in general. After discussion, Member States experts were of the opinion that identification should be based on FT-IR combined with Thermo Gravimetric (TG) analysis and solubility properties of the fibre. The microscopic analysis can be useful but identification cannot be based just on this technique. For the identification of melamine in mixtures, FT-IR measurements should be performed after chemical dissolution of the soluble fibre.

The value of 7.00 was established for the agreed allowance of melamine fibre and approved during the technical meeting of national experts on textile labelling held in Ispra on 21st April 2008.

The normal pre-treatment, described in the Directive 96/73/EC, can be applied to the new fibre. In addition, same results of mass loss were obtained both using a traditional Soxhlet and an automatic extractor (Soxtec).

Tests results showed that melamine is insoluble with all methods of the Directive 96/73/EC. The correction factors *d* for all the chemical methods described in the Directive (except method 12) were calculated based on the analyses of samples of pure melamine. The values were generally equal to 1.01, except in the case of method 3 and 10, for which *d* value was 1.00.

On 19th September 2007, during the 7th meeting of the European Network of National Experts on Textile Labelling, it was agreed to confirm with a ring trial the correction factors *d* of melamine for five methods: method 3 for binary mixtures with viscose or cotton; method 4 for mixtures with polyamide; method 7 dissolving viscose and cotton; method 8 for mixtures with modacrylic and method 14 dissolving polyester. The inter-laboratory trial results established the value of 1.01 as melamine correction factor for methods 3, 4, 8 and 14. The *d* value calculated in the ring trial for method 7 was 1.07, however the repeatability and reproducibility limits calculated were very poor and the data could not be considered as being part of a normal distribution. One

explanation of such poor results could be the very strong influence of temperature on the solubility properties of melamine with this method. For these reasons, in the 8th meeting of the Network, on 21th April 2008, it was decided that method 7 is not applicable to blends containing melamine.

The petitioner proposed three quantification methods: the first one based on a hot sodium hypochloride solution in which melamine was supposed to be insoluble; the second one foresaw a hot sulphuric acid solution to dissolve melamine and it was suggested, in conjunction with the first method, to determine the melamine content of blends containing cellulosic fibres along with other fibres which are not soluble in the reagent; the third one was suggested to dissolve and quantify melamine when blended with aramid fibres using a hot formic acid solution. Contact time and temperature were three hours at 90 °C for all the three methods.

First of all, the JRC evaluated the solubility properties of melamine and other common fibres with all the methods and a modified version of method 2 of Directive 96/73/EC. Then the influence of contact time (1-3 hours) and temperature (80-95 °C) on the quantification of both pure melamine and binary mixtures was investigated. In the case of the hot formic acid, the influence of the acid concentration was also studied. In addition performances of the hot formic and sulphuric acid methods were compared in the quantification of binary blends melamine/aramid.

Melamine was found to be partially soluble in hot sodium hypochloride solution both at the concentration proposed by the petitioner (6.15 %) and the one prescribed for method 2.

A change in contact time from three to one hour did not influence results obtained with the hot formic acid method either on pure melamine or on binary mixtures. On the contrary, for the hot sodium hypochloride method, different results were obtained by changing this parameter and the same was true in the case of three out of six binary blends analysed with the hot sulphuric acid method.

The effect of temperature was checked using a contact time of one hour. This parameter had a strong influence on the solubility properties of melamine and consequently on all the methods proposed by Basofil and the modified version of method 2. As a conclusion, the temperature should be carefully controlled between 90 ± 2 °C. In fact a larger range, such as 90 ± 5 °C, did not provide comparable results.

Experiments performed at 90 °C with one hour contact time showed that the concentration of the formic acid solution can be varied in the range 87-98-100 % without influencing results.

The comparison between performances of the two methods with hot acid solutions evidenced some difficulties in the case of the method with sulphuric acid to completely dissolve melamine, using one and three hours contact time.

Based on the experimental data, the following conclusions were agreed with experts from Member States concerning the three methods proposed by Basofil: first, the method with sodium hypochloride at 90°C was deemed not applicable due to partial solubility of melamine, which was supposed to be insoluble; second, the hot formic acid method was considered adequate and applicable in the case of binary mixtures of melamine with aramid and cotton; third, the hot sulphuric acid method was judged not adequate as results obtained with this method were statistically different compared to those obtained with the hot formic acid method and the reagent showed difficulties in completely dissolving melamine. Moreover, experts were of the opinion that it was not necessary to have two methods to dissolve melamine and they considered that the hot formic acid method was sufficient.

During the 7th meeting of the European Network of National Experts on Textile Labelling, on 19th September 2007, a consensus was reached on the need to validate the new method based on hot formic acid and to confirm the correction factors *d* for aramid (meta and para) and cotton. Three samples, two binary blends with aramid and one with cotton, were chosen for the validation exercise, plus three samples of pure cotton, meta and para aramid. The JRC organised the ring trial with the participation of seventeen European laboratories, in accordance with the rules laid down in ISO 5725 (1994), and discussed results during the 8th meeting of the Network. The validation was successful. The precision of the test method, which should be quoted as a percentage by mass, was expressed as repeatability and reproducibility limits. Results were 0.74 % and 1.77 %, respectively. The correction factors *d* for aramid and cotton were evaluated in the same context and the established values are 1.02 for both fibres.

The name melamine was chosen for the new fibre in agreement with experts from Member States, as it fulfils the criteria set up in 2002 by Commission and technical experts working group on textile labelling. In fact, according to these criteria, a

generic name should not link the fibre to a specific manufacturer, it should be free of rights and it should inform consumers about characteristics of the fibre.

On the basis of the experimental results and of discussions with representative experts during the meeting of the European network of national experts on textile labelling held in Ispra on 21st April 2008, the definition agreed and proposed for melamine is: "*fibre formed of at least 85 % by mass of cross-linked macromolecules made up of melamine derivatives*".

5. References

[1] Directive 96/74/EC of the European Parliament and of the Council of 16 December 1996 on textile names *(Official Journal L032 of 3.2.1997 p. 0038-0055),* http://europa.eu.int/comm/enterprise/textile/intlmarket.htm.

[2] Directive 96/73/EC of the European Parliament and of the Council of 16 December 1996 on certain methods for the quantitative analysis of binary textile fibre mixtures *(Official Journal L032 of 3.2.1997 p. 0001-0037),* http://europa.eu.int/comm/enterprise/textile/intlmarket.htm.

[3] ISO 1833 (2006) Textile – Quantitative chemical analysis. Part 1: General principles of testing.

[4] UNI 9213 (1989) Determination of commercial mass.

[5] UNI 8048 (1980) Determination of dry content for chemical and/or physical measurements.

[6] ISO 5725 (1994) Accuracy (trueness and precision) of measurement methods and results, Part 1-6.

[7] Horwitz, W. (1995) IUPAC: Protocol for the design, conduct and interpretation of method performance studies, *Pure & Applied Chem.*, **67**, 331-343.

[8] Stevenson, C. L., The statistic of measurements, University of Richmond, Chemistry 300 (2000).

[9] P. Piccinini, E. Buriova, Validation of a new quantitative method for the analysis of fibre blends containing melamine and aramid or cotton, 2008, EUR EN.

[10] Directive 73/44/EEC of the European Parliament and of the Council of 23 February 1973 on the approximation of the laws of the Member States relating to the quantitative analysis of ternary fibre mixtures *(Official Journal L083 of 30.3.1973 p. 0001-0019),* http://europa.eu.int/comm/enterprise/textile/intlmarket.htm.

Annex I

Analytical methods proposed by the applicant

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

ATTACHMENT #2:

TITLE: BASOFIL FIBER ANALYSIS IN BLENDS WITH CELLULOSIC, MODACRYLIC AND POLYESTER FIBERS - BLEACH DISSOLUTION PROCEDURE

LAB METHOD NO: **BAS-0002-03** TOTAL PAGES: 3

AUTHORED BY: A.C.HANDERMANN DATE AUTHORED: October 14, 2003

REVISION BY: A.C.HANDERMANN REVISION DATE: January 17, 2006

LOCATION: BASOFIL FIBERS LLC SITE: ENKA, NC

1. PURPOSE
2. SCOPE
3. PRINCIPLE
4. PRECISION/BIAS
5. SAFETY
6. APPARATUS
7. REAGENTS
8. PREPARATION
9. PROCEDURE
10. CALCULATION

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

1. PURPOSE: This method is used, in conjunction with method BAS-0003-03, to determine the Basofil fiber content of fiber blends containing cellulosic fibers, along with modacrylic, polyester or other fibers which are not soluble in hot 6.15% sodium hypochlorite solution.

2. SCOPE: To determine the cellulosic fiber content (Visil, Lenzing FR, Tencel, cotton, rayon or other cellulosic fiber) in a fiber blend by dissolving with hot 6.15% sodium hypochlorite solution.

3. PRINCIPLE: A known amount of blended fiber sample is heated in a solution of 6.15% sodium hypochlorite at 90°C for 3 hours on an oscillating hotplate. The spent liquid is decanted, the sample washed with tap water and the residue dried and weighed to determine percent solubles.

4. PRECISION: Cellulosic fibers are 100% soluble in hot 6.15% sodium hypochlorite solution.

BIAS: Basofil, modacrylic and polyester fibers are completely insoluble in hot 6.15% sodium hypochlorite.

5. SAFETY: Hot sodium hypochlorite is very corrosive and must be handled with care. Wear safety glasses, chemical and heat resistant gloves and a lab coat during the test. CAUTION: Do not mix NaOCl with any amine containing material. Dispose spent solution by washing down the drain with plenty of water.

6. APPARATUS:

6.1 "Large Mouth" Erlenmeyer flasks (500 mL).
6.2 Oscillating hot plate.
6.3 Analytical balance, ± .0001 gram.
6.4 Tweezers - 10 inch.
6.5 Desiccator containing 8 mesh drierite.
6.6 Recirculating hot air drying oven set at 105°C.

7. REAGENTS:

7.1 Sodium hypochlorite - "Ultra Clorox" – 6.15% NaOCl.

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

7.2 Tap water.

8. PREPARATION:

8.1 Dry 6 - 8 grams of fiber sample at 105°C for 1 hour and cool to room temperature in a desiccator prior to weighing.

9. PROCEDURE (perform in triplicate):

9.1 Weigh about 2 grams of desiccated fiber sample to the nearest 0.0001 g and place into a 500 mL Erlenmeyer flask.

9.2 Add 200 mL of 6.15% sodium hypochlorite to the flask containing the sample.

9.3 Place flask on an oscillating hot plate which has been set to heat liquids to 90°C for 3 hours.

9.4 Remove flask and decant spent solution by washing down the drain with plenty of water, making sure none of the fiber residue is lost.

9.5 Refill flask about halfway with tap water and shake contents. Decant wash water into sink, being careful to ensure that all residual fiber remains in flask.

9.6 Repeat step 9.5 at least 4-5 times, ensuring that rinse water is completely clear.

9.7 After final rinse, carefully pour off remaining rinse water and remove residual fiber. Rinse fiber residue with tap water and squeeze excess water from fiber, making sure no residual fiber is lost.

9.8 Place damp fiber residue in oven at 105°C for 1 hour to dry thoroughly.

9.9 Desiccate and cool sample and filter paper to room temperature and weigh to nearest 0.0001 g.

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

10. CALCULATION:

10.1 The following is the formula for determining the percent solubility of cellulosic fibers in hot 6.15% sodium hypochlorite.

*% Soluble = (A – B) * 100 / A*

A = original dried/desiccated sample, grams

B = dried/desiccated residue, grams

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

ATTACHMENT #3:

TITLE: BASOFIL FIBER ANALYSIS IN BLENDS WITH CELLULOSIC, MODACRYLIC AND POLYESTER FIBERS - SULFURIC ACID DISSOLUTION PROCEDURE

LAB METHOD NO: **BAS-0003-03** TOTAL PAGES: 3

AUTHORED BY: A.C.HANDERMANN DATE AUTHORED: October 14, 2003

REVISION BY: A.C.HANDERMANN REVISION DATE: January 17, 2006

LOCATION: BASOFIL FIBERS LLC SITE: ENKA, NC

1. PURPOSE
2. SCOPE
3. PRINCIPLE
4. PRECISION/BIAS
5. SAFETY
6. APPARATUS
7. REAGENTS
8. PREPARATION
9. PROCEDURE
10. CALCULATION

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

1. PURPOSE: This method is used, in conjunction with method BAS-0002-03, to determine the Basofil fiber content of fiber blends containing cellulosic fibers, along with modacrylic, polyester or other fibers which are not soluble in hot 50 wt% sulfuric acid.

2. SCOPE: To determine the combined Basofil and cellulosic fiber contents (Visil, Lenzing FR, Tencel, cotton, rayon or other cellulosic fiber) in a fiber blend by dissolving with hot 50 wt% sulfuric acid.

3. PRINCIPLE: A known amount of blended fiber sample is heated in a solution of 50 wt% sulfuric acid at 90°C for 3 hours on an oscillating hotplate. The spent liquid is decanted, the sample washed with tap water and the residue dried and weighed to determine percent solubles.

4. PRECISION: Basofil and cellulosic fibers are 100% soluble in hot 50 wt% sulfuric acid.

BIAS: Modacrylic and polyester fibers are completely insoluble in hot 50 wt% sulfuric acid.

5. SAFETY: Hot sulfuric acid is very corrosive and must be handled with care. Wear safety glasses, chemical and heat resistant gloves and a lab coat during the test. CAUTION: Dispose of spent solution by washing down the drain with plenty of water.

6. APPARATUS:

6.1 "Large Mouth" Erlenmeyer flasks (500 mL).
6.2 Oscillating hot plate.
6.3 Analytical balance, ± .0001 gram.
6.4 Tweezers - 10 inch.
6.5 Desiccator containing 8 mesh drierite.
6.6 Recirculating hot air drying oven set at 105°C.

7. REAGENTS:

7.1 50 wt% Sulfuric Acid

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

7.2 Tap water

8. PREPARATION:

8.1 Dry 6 - 8 grams of fiber sample at 105°C for 1 hour and cool to room temperature in a desiccator prior to weighing.

9. PROCEDURE (perform in triplicate):

9.1 Weigh about 2 grams of desiccated fiber sample to the nearest 0.0001 g and place into a 500 mL Erlenmeyer flask.

9.2 Add 200 mL of 50 wt% sulfuric acid to the flask containing the sample.

9.3 Place flask on an oscillating hot plate which has been set to heat liquids to 90°C for 3 hours.

9.4 Remove flask and decant spent solution by washing down the drain with plenty of water, making sure none of the fiber residue is lost.

9.5 Refill flask about halfway with tap water and shake contents. Decant wash water into sink, being careful to ensure that all residual fiber remains in flask.

9.6 Repeat step 9.5 at least 4-5 times, ensuring that rinse water is completely clear.

9.7 After final rinse, carefully pour off remaining rinse water and remove residual fiber. Rinse fiber residue with tap water and squeeze excess water from fiber, making sure no residual fiber is lost.

9.8 Place damp fiber residue in oven at 105°C for 1 hour to dry thoroughly.

9.9 Desiccate and cool sample and filter paper to room temperature and weigh to nearest 0.0001 g.

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

10. CALCULATION:

10.1 The following is the formula for determining the combined percent solubility of Basofil and cellulosic fibers in hot 50 wt% sulfuric acid.

*% Soluble = (A – B) * 100 / A*

A = original dried/desiccated sample, grams

B = dried/desiccated residue, grams

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

ATTACHMENT #4:

TITLE: BASOFIL FIBER ANALYSIS IN BLENDS WITH ARAMID FIBERS – FORMIC ACID DISSOLUTION PROCEDURE

LAB METHOD NO: **CA-G1011-92** TOTAL PAGES: 3

AUTHORED BY: C. B. JUSTICE DATE AUTHORED: August 4, 1992

REVISED BY: A. C. HANDERMANN REVISION DATE: January 17, 2006

LOCATION: BASOFIL FIBERS LLC SITE: ENKA, NC

SUMMARY OF PROCEDURE:

1. PURPOSE
2. SCOPE
3. PRINCIPLE
4. PRECISION
5. SAFETY
6. APPARATUS
7. REAGENTS
8. PREPARATION
9. PROCEDURE
10. CALCULATION
11. INTERPRETATION OF RESULTS

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

1. PURPOSE: To determine the percent solubility of Basofil fiber when blended with aramid fibers utilizing hot formic acid as a solvent.

2. SCOPE: The method is suitable for Basofil fiber blends with Kevlar, Twaron, Nomex, Conex and any other fibers that are insoluble or only very slightly soluble in 90% formic acid.

3. PRINCIPLE: A known amount of sample is heated in 90% formic acid at 90°C for 3 hours, collected through a filtration funnel, dried and weighed to determine solubles.

4. PRECISION: Basofil is 100% soluble in hot formic acid. Very slightly soluble are Kevlar = 1.5%, ± 0.5; Nomex = 2.5%, ± 0.5 and Conex = 2.5%, ± 0.5.

5. SAFETY: Hot formic acid is very corrosive and must be handled with care. Wear safety glasses, chemical and heat resistant gloves and lab coat during the test. Dispose of spent solvent by neutralizing and washing down the drain with plenty of water.

6. APPARATUS:

- 6.1 Large mouth Erlenmeyer flasks (500 mL).
- 6.2 Oscillating hot plate.
- 6.3 Fritted glass filter funnels (coarse).
- 6.4 Analytical balance, ±0.0001 g.
- 6.5 Graduated cylinder (100 mL).
- 6.6 Tweezers - 10 inch.
- 6.7 Desiccator containing 8 mesh drierite.
- 6.8 Drying oven set at 105°C.

7. REAGENTS:

- 7.1 Formic acid, 90% reagent grade.
- 7.2 Distilled water.
- 7.3 Methanol, reagent grade.

8. PREPARATION:

- 8.1 Dry sample at 105°C for 1 hour and desiccate prior to weighing.

Basofil Fibers, LLC
4824 Parkway Plaza Boulevard
Suite 250
Charlotte, NC 28217
phone 704.423.2418
fax 704.423.2261

9. PROCEDURE:

9.1 Add 200 mL 90% Formic Acid to a 500 mL Erlenmeyer flask.

9.2 Weigh 2g of desiccated sample to 0.0001 gm, record weight and place into 500 mL Erlenmeyer flask with 90% formic acid.

9.3 Place flask on 90°C oscillating hot plate for 3 hours.

9.4 Cool to room temperature.

9.5 Filter through a dry coarse fritted filter funnel.

9.6 Immediately rinse with 50 mL formic acid (90%).

9.7 Rinse thoroughly with methanol (3 times with 50 mL).

9.8 Air dry and place in 105°C circulating oven for 2 hours.

9.9 Allow to cool in desiccator and reweigh to 0.0001 gram.

10. CALCULATIONS:

10.1 Determination of % Solubles.

$$\% \text{ Solubles} = (A - B) / A * 100$$

A = sample weight recorded in 9.2 in grams.
B = sample weight recorded in 9.9 in grams.

10.2 Determination of % Basofil fiber.

$$\% \text{ Basofil} = (((A - B) / A) - (X * (1 - (A - B) / A))) * 100$$

X = 0.015 when blending fiber is Kevlar
X = 0.025 when blending fiber is Nomex or Conex

Annex II

Microscopic analysis

100% melamine (sample 136)

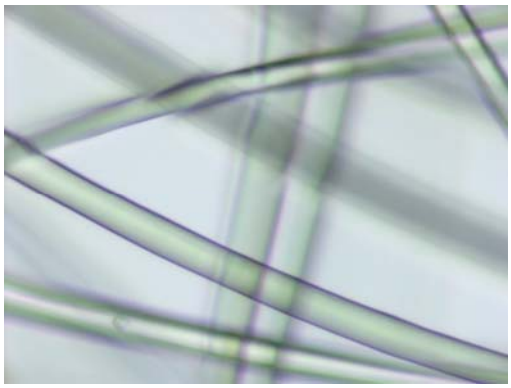


Fig. 1: Sample **136**, longitudinal view, 630X.

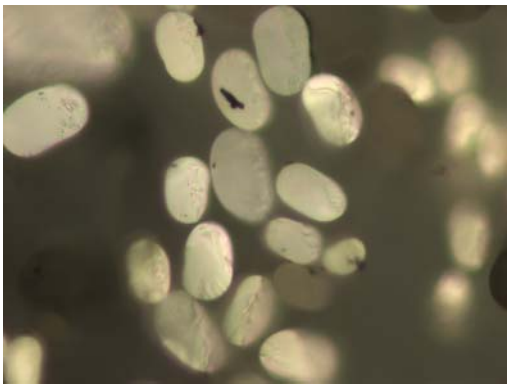


Fig. 2: Sample **136**, cross-section, 630X.

100% melamine (sample 137)

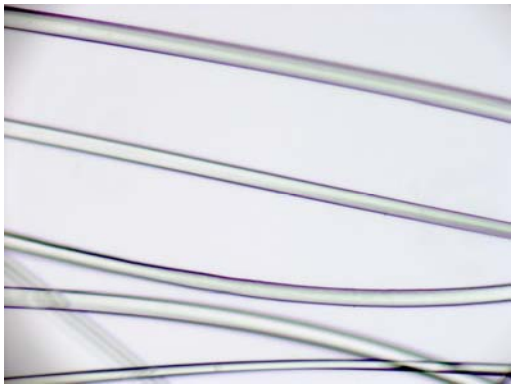


Fig. 3: Sample **137,** longitudinal view, 400X.

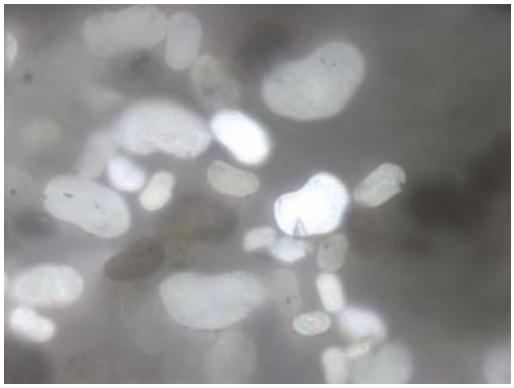


Fig. 4: Sample **137**, cross-section, 630X.

40 % melamine - 60 % m-aramid (Nomex) (sample 138)

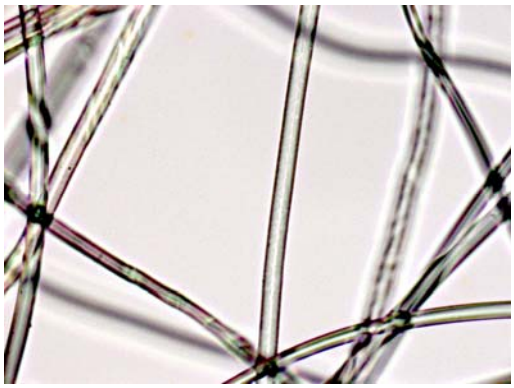


Fig. 5: Sample **138,** longitudinal view, 200X.

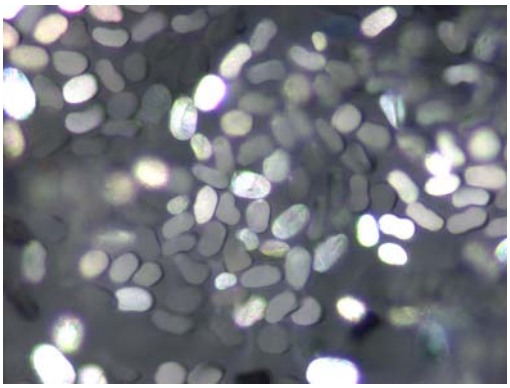


Fig. 6: Sample **138**, cross-section, 400X.

40 % melamine - 60 % m-aramid (Nomex) (sample 139)

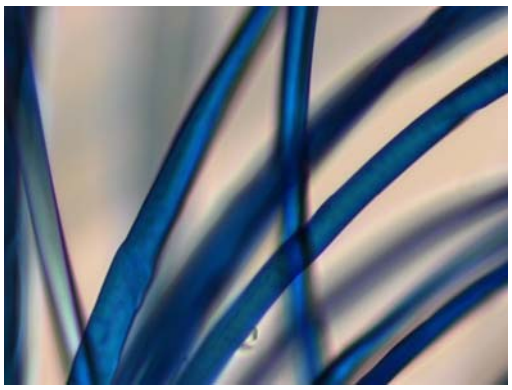


Fig. 7: Sample **139,** longitudinal view, 400X.

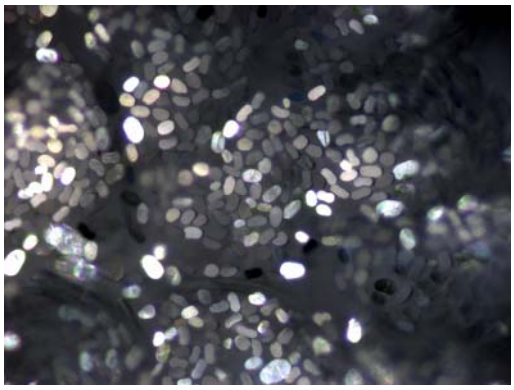


Fig. 8: Sample **139**, cross-section, 200X.

50 % melamine - 50 % m-aramid (Nomex) (sample 140)

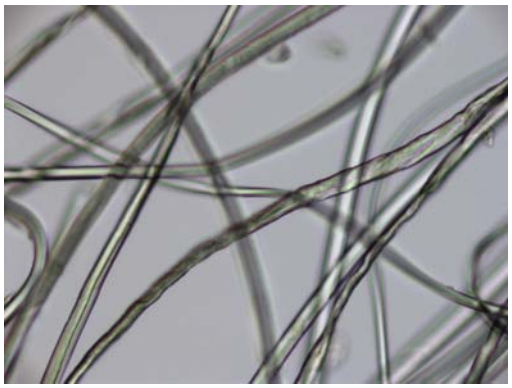


Fig.9: Sample **140,** longitudinal view, 200X.

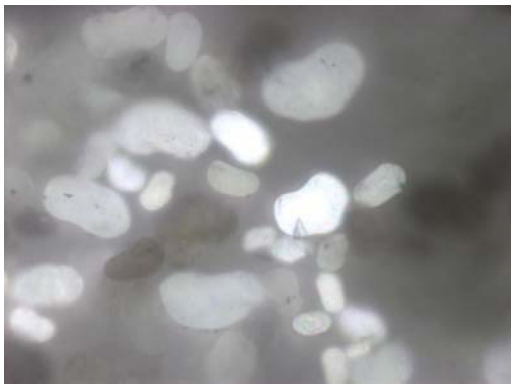


Fig. 10: Sample **140**, cross-section, 630X.

40 % melamine - 60 % p-aramid (Kevlar) (sample 141)

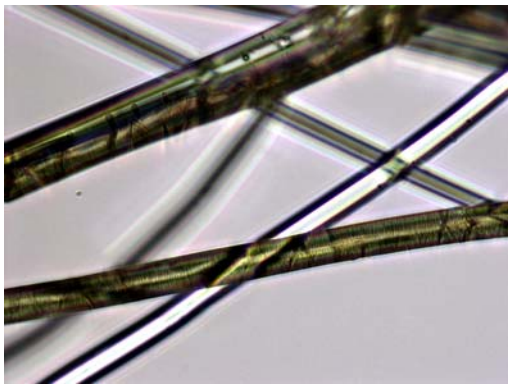


Fig. 11: Sample **141,** longitudinal view, 400X.

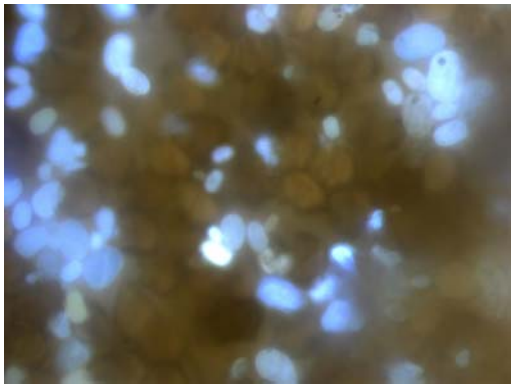


Fig. 12: Sample **141**, cross-section, 400X.

40 % melamine - 60 % p-aramid (Kevlar) (sample 142)

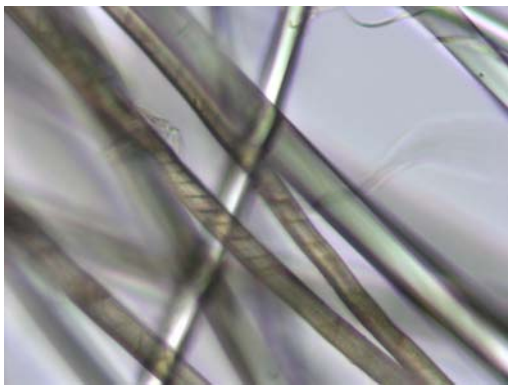


Fig. 13: Sample **142,** longitudinal view, 400X.

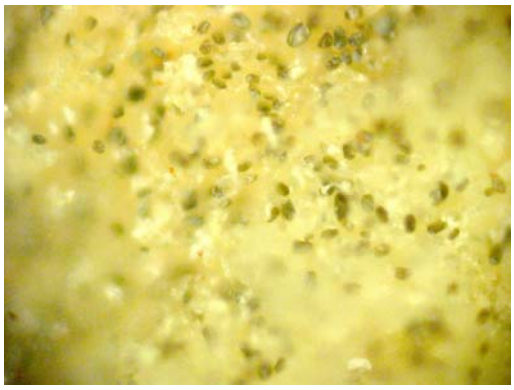


Fig. 14: Sample **142**, cross-section, 200X.

25 % melamine - 75 % m-aramid (Conex) (sample 143)

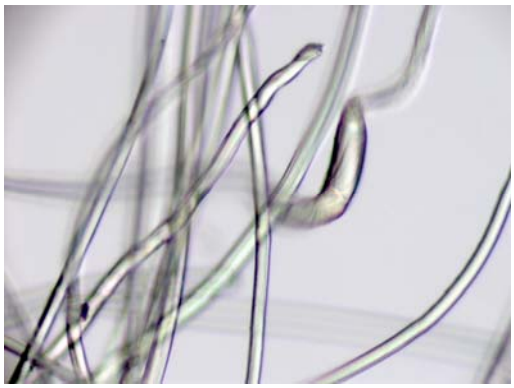


Fig. 15: Sample **143,** longitudinal view, 200X.

Fig. 16: Sample **143**, cross-section, 630X.

17 % melamine - 17 % modacrylic – 66% lm polyester (sample 144)

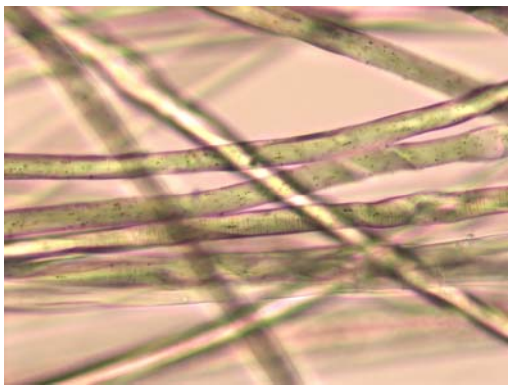


Fig. 17: Sample **144,** longitudinal view, 400X.

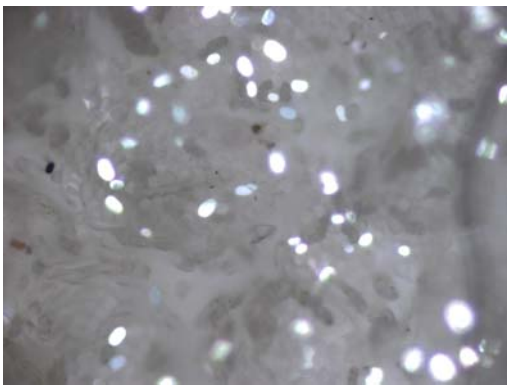


Fig. 18: Sample **144**, cross-section, 200X.

12 % melamine – 30% lyocell -34 % modacrylic – 34% lm polyester (sample 145)

Fig. 19: Sample **145,** longitudinal view, 200X.

Fig. 20: Sample **145**, cross-section, 200X.

melamine - viscose (sample 172)

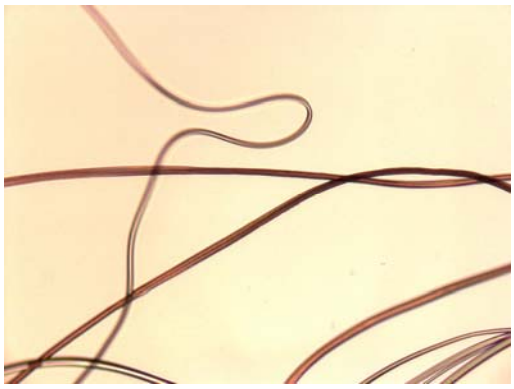


Fig.21: Sample **172,** longitudinal view, 100X.

Fig. 22: Sample **172**, cross-section, 630X.

melamine - Visil (sample 173)

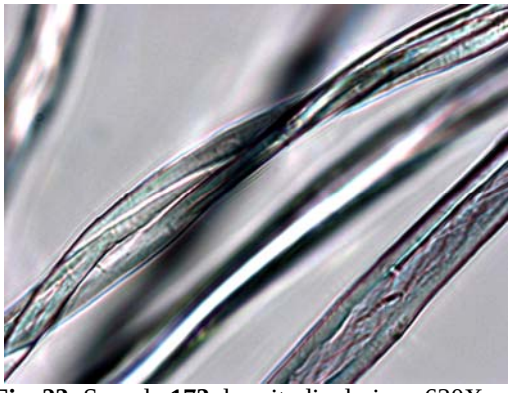


Fig. 23: Sample **173**, longitudinal view, 630X.

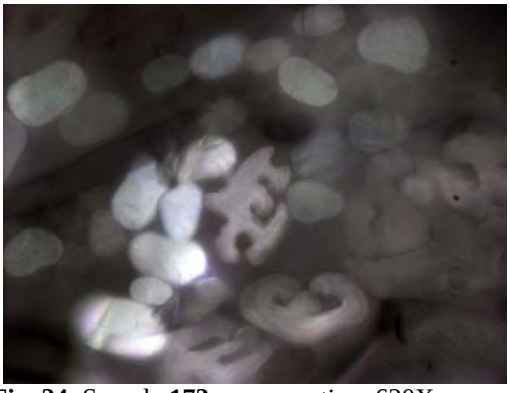


Fig. 24: Sample **173**, cross-section, 630X.

melamine - polyester (sample 174)

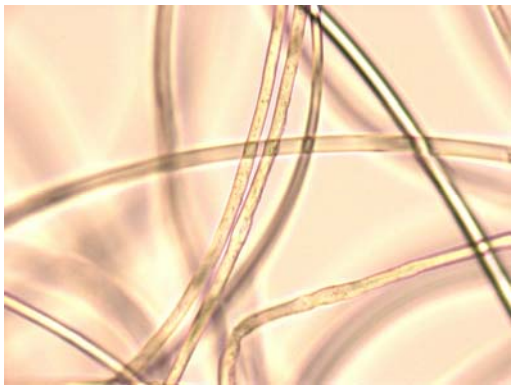


Fig. 25: Sample **174**, longitudinal view, 200X.

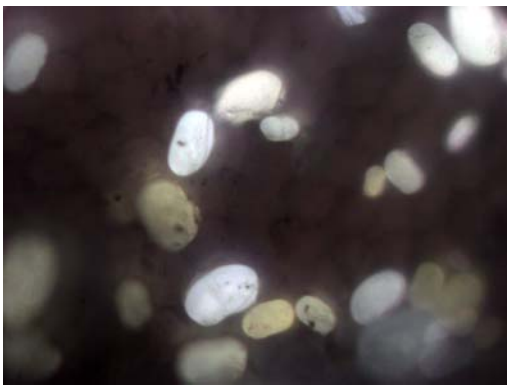


Fig. 26: Sample **174**, cross-section, 630X.

melamine - cotton (sample 175)

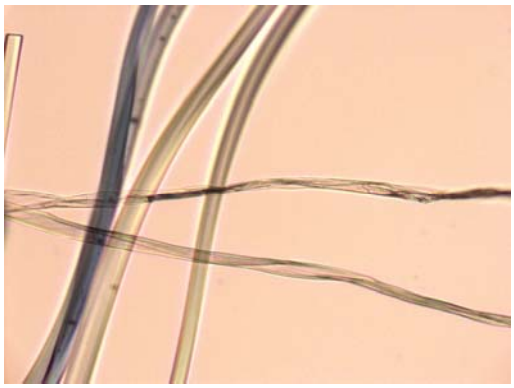


Fig. 27: Sample **175**, longitudinal view, 200X.

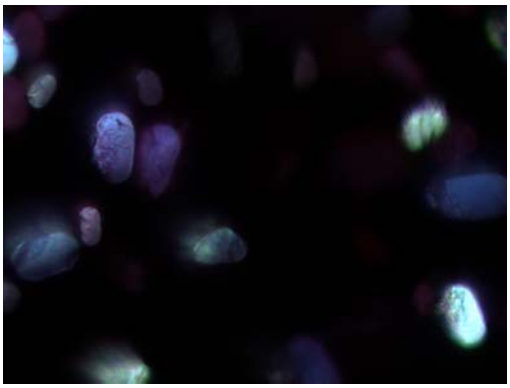


Fig. 28: Sample **175**, cross-section, 630X.

melamine - viscose (sample 176)

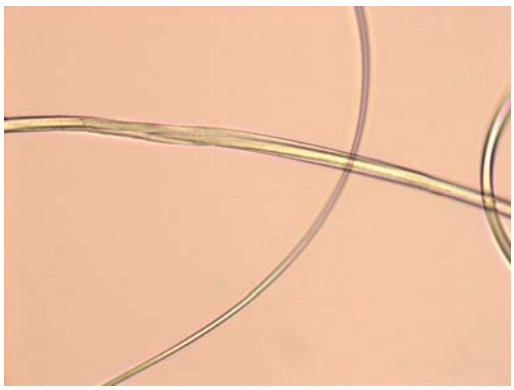


Fig. 29: Sample **176**, longitudinal view, 200X.

Fig. 30: Sample **176**, cross-section, 630X.

melamine – m-aramid (Nomex) (sample 177)

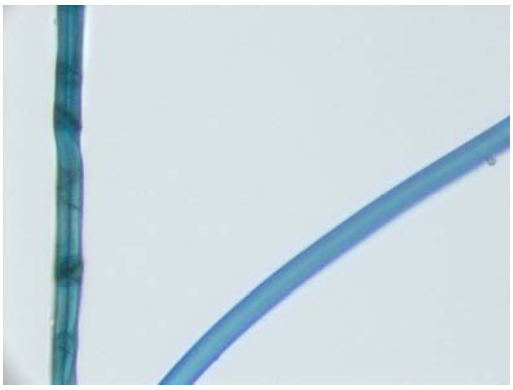


Fig. 31: Sample **177**, longitudinal view, 400X

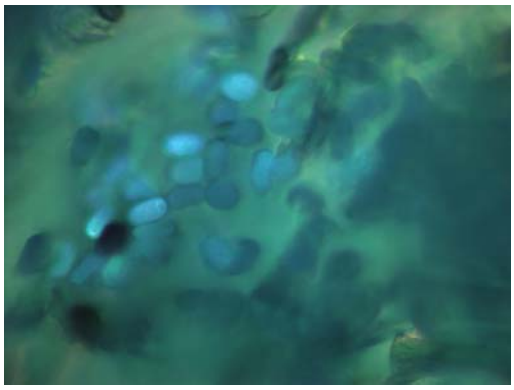


Fig. 32: Sample **177**, cross-section, 200X.

melamine – p-aramid (Kevlar) (sample 178)

Fig. 33: Sample **178**, longitudinal view, 200X

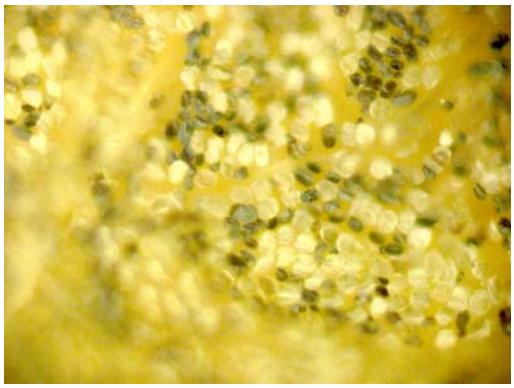


Fig. 34: Sample **178**, cross-section, 200X.

Annex III

Spectroscopic analysis
FT-IR

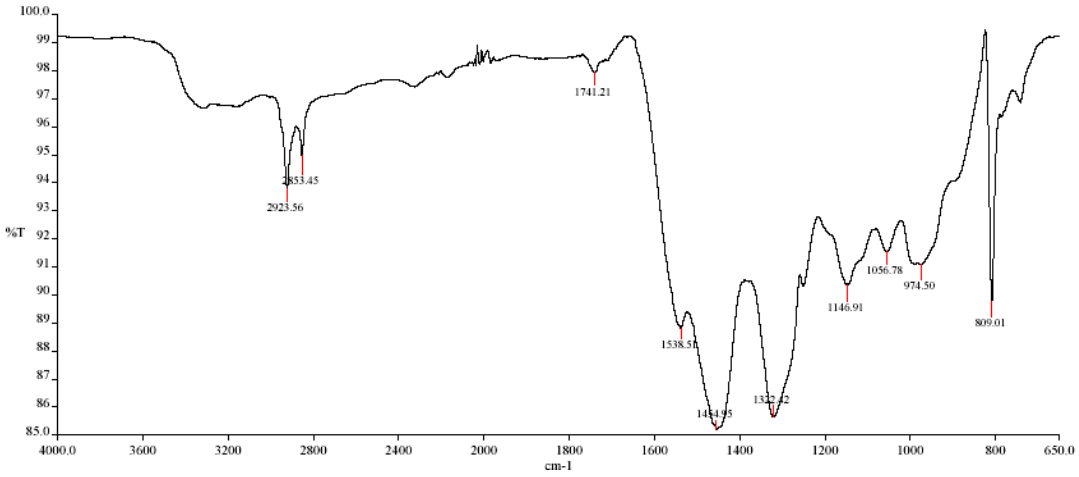


Fig.1: FT-IR spectrum of untreated melamine (sample **136**).

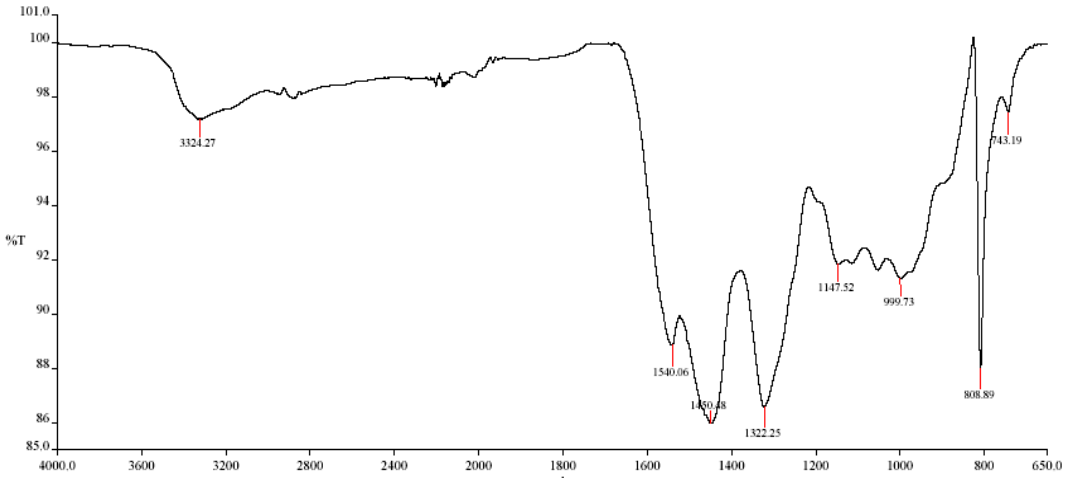


Fig.2: FT-IR spectrum of melamine pre-treated in Soxhlet apparatus (sample **136**).

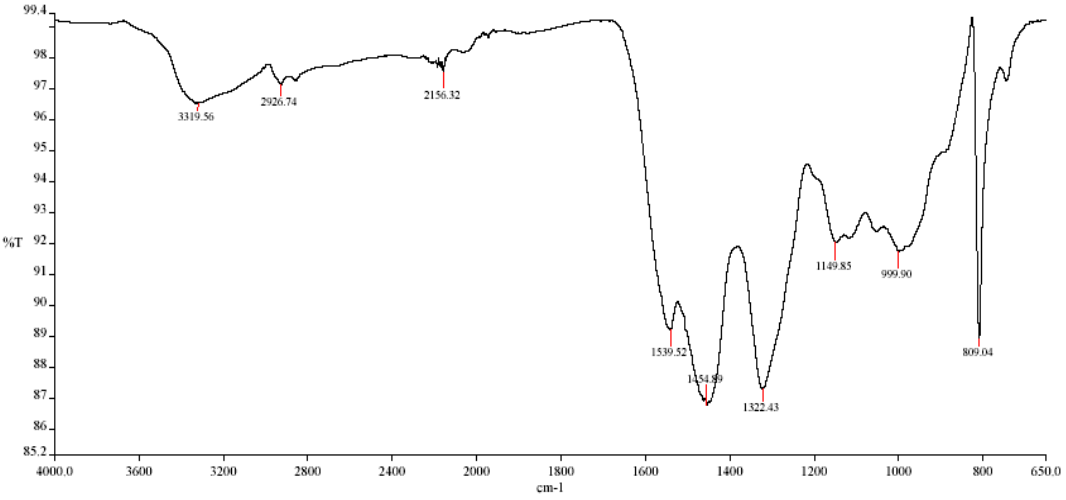


Fig.3: FT-IR spectrum of melamine pre-treated in Soxtec apparatus (sample **136**).

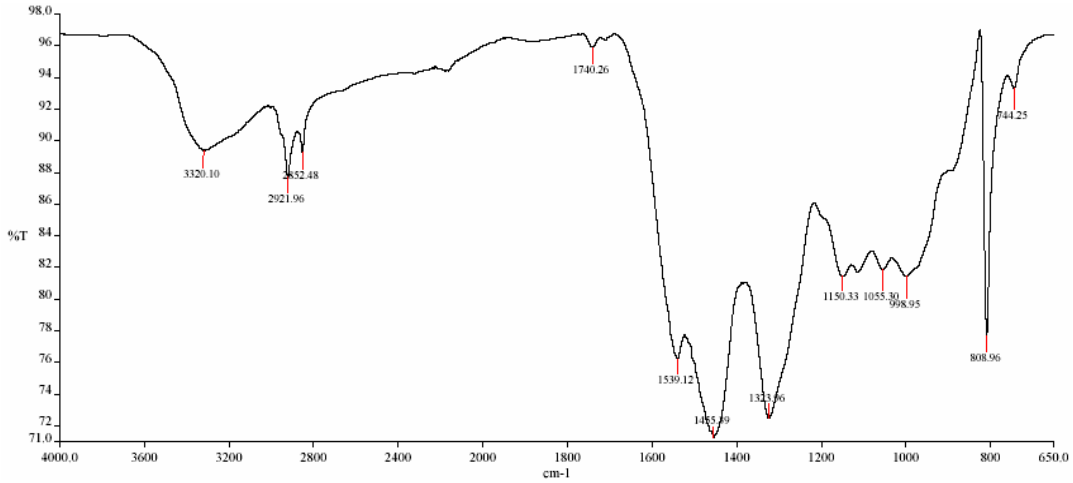


Fig.4: FT-IR spectrum of untreated melamine (sample **137**).

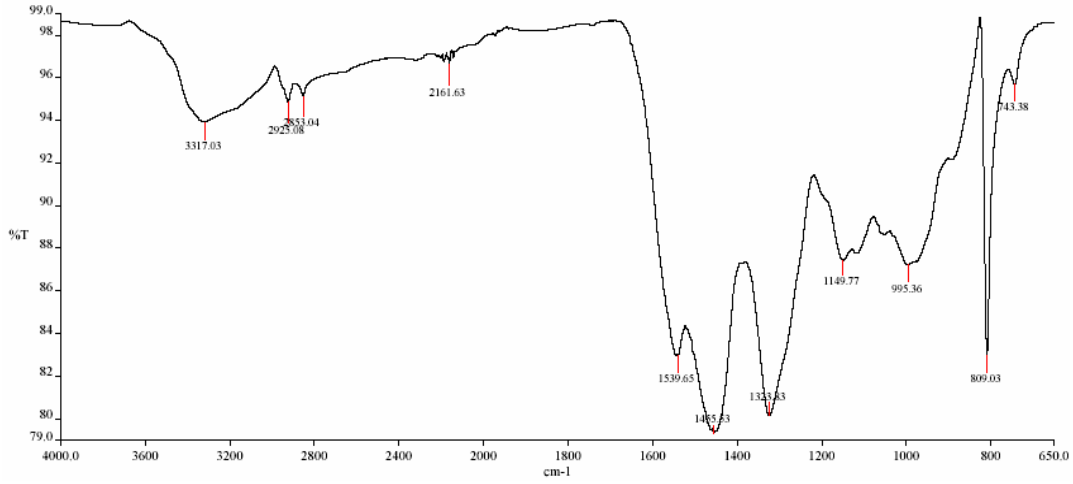


Fig.5: FT-IR spectrum of melamine pre-treated in Soxhlet apparatus (sample **137**).

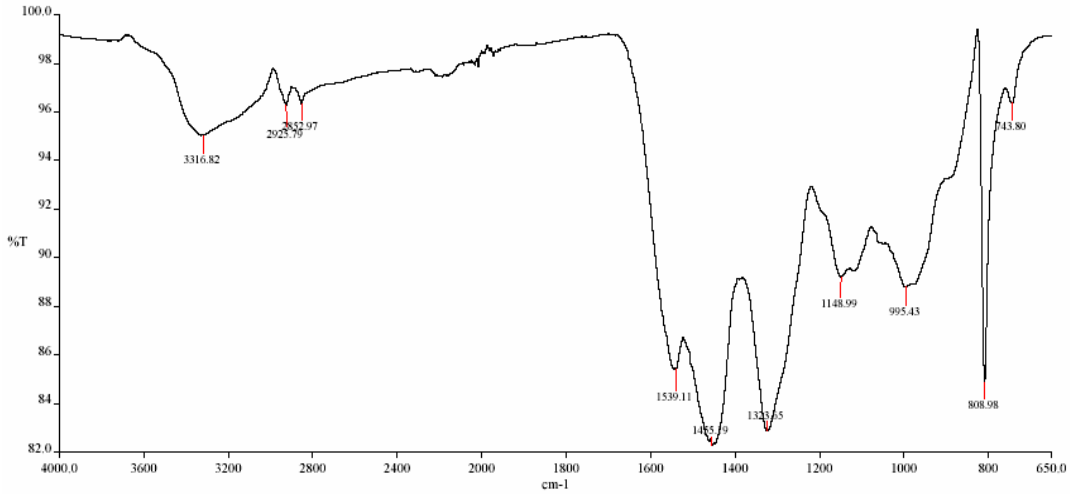


Fig.6: FT-IR spectrum of melamine pre-treated in Soxtec apparatus (sample **137**).

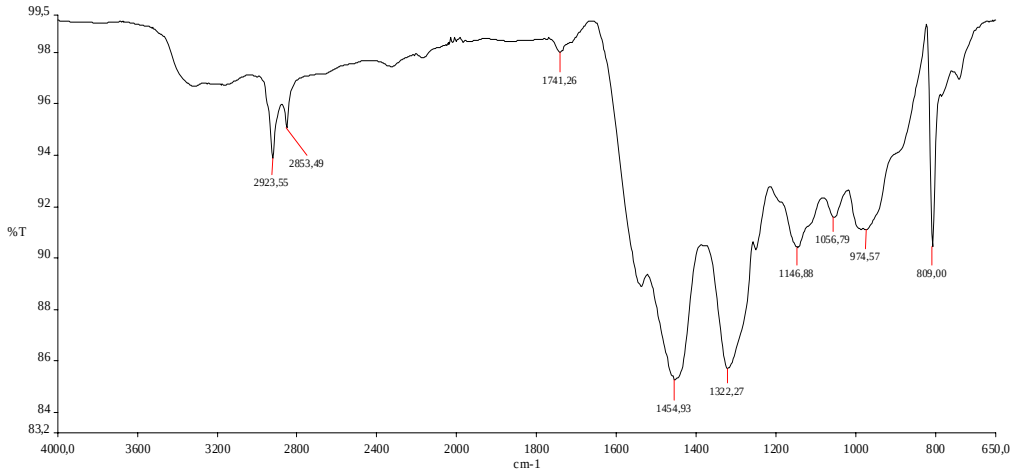


Fig.7: FT-IR spectrum of untreated melamine (sample **171**).

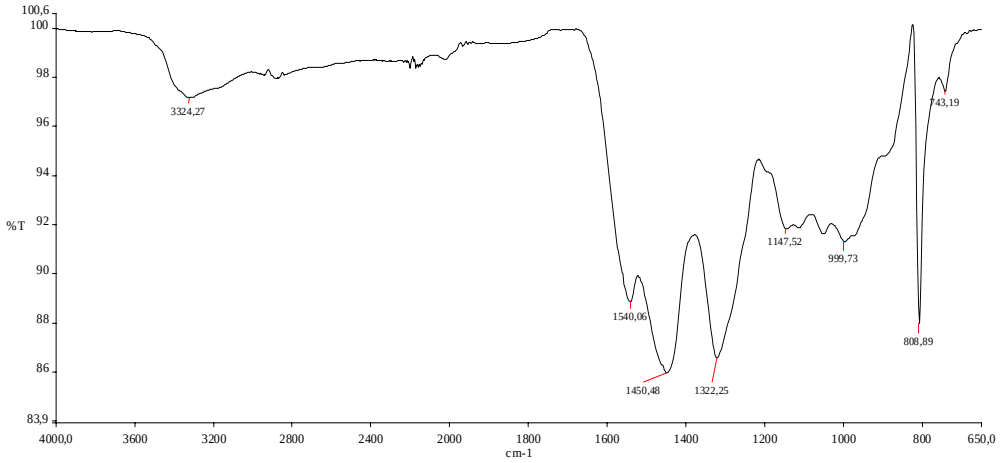


Fig.8: FT-IR spectrum of melamine pre-treated in Soxhlet apparatus (sample **171**).

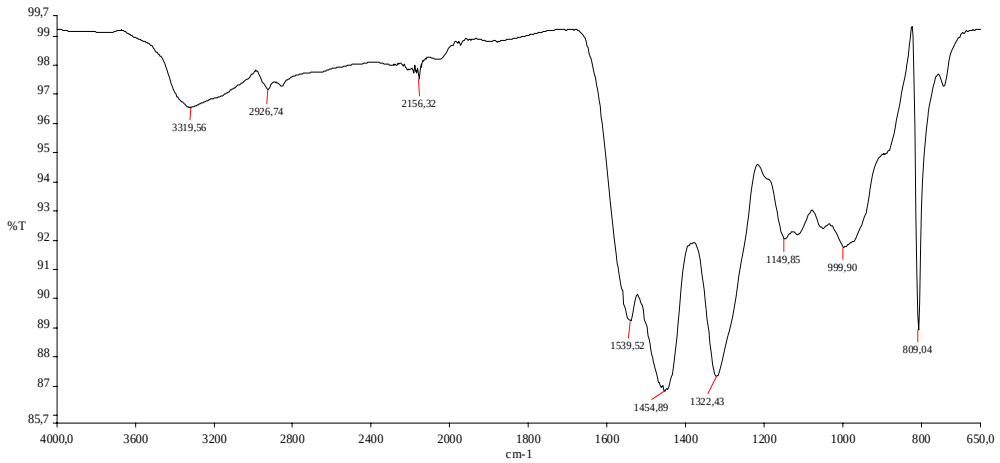


Fig.9: FT-IR spectrum of melamine pre-treated in Soxtec apparatus (sample **171**).

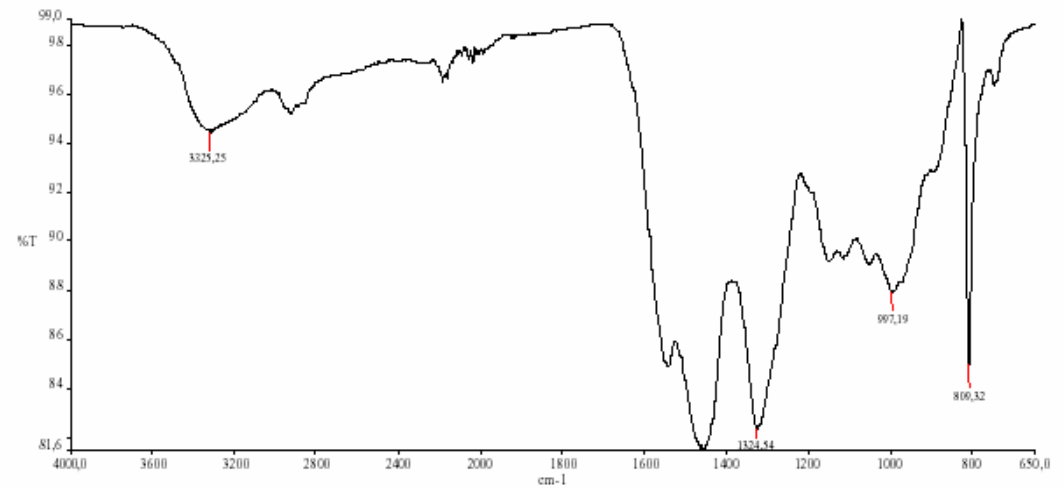


Fig.10: FT-IR spectrum of untreated melamine (sample **179**).

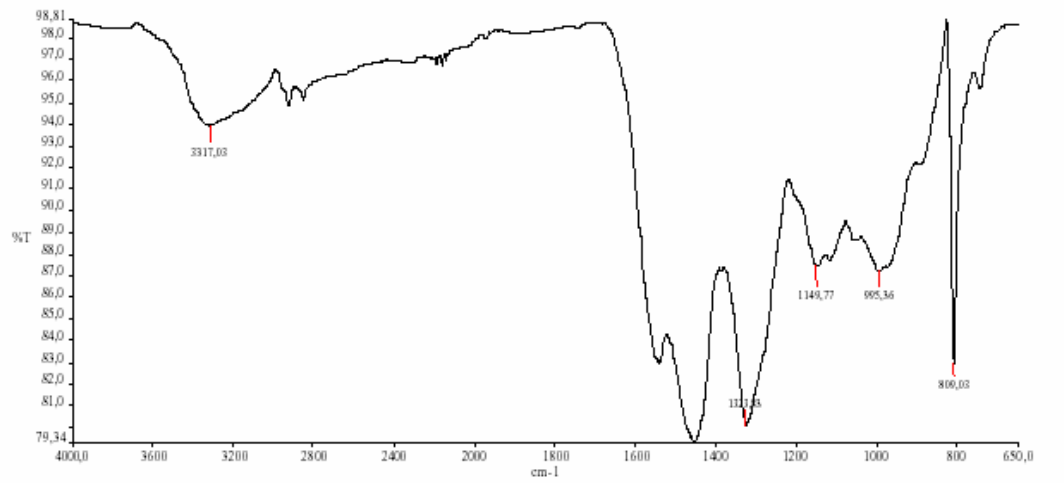


Fig.11: FT-IR spectrum of melamine pre-treated in Soxhlet apparatus (sample **179**).

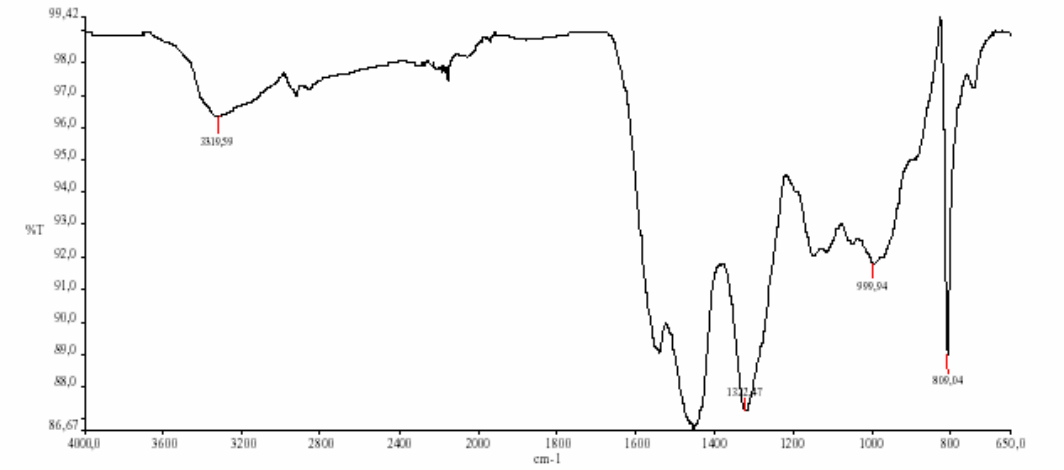


Fig.12: FT-IR spectrum of melamine pre-treated in Soxtec apparatus (sample **179**).

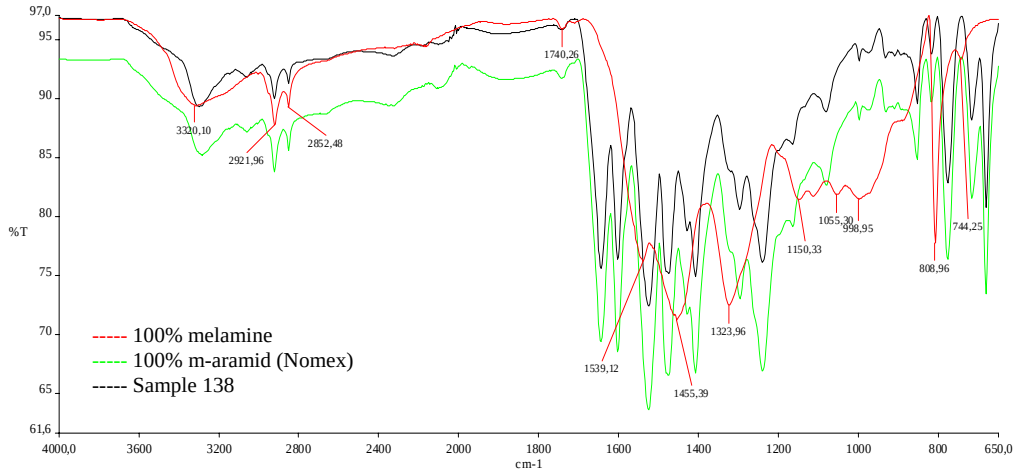


Fig.13: FT-IR spectrum of sample **138 (**binary mixture m-aramid (Nomex)/melamine) and of the pure component fibres.

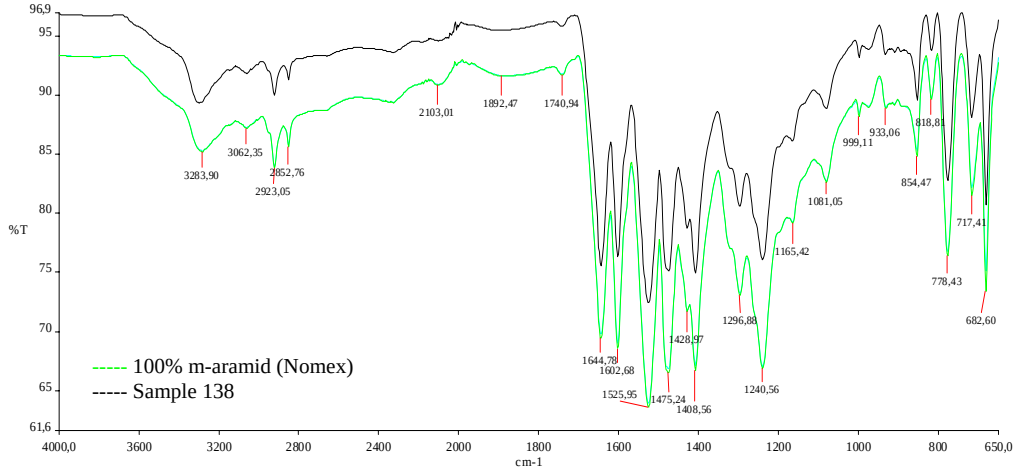


Fig.14: FT-IR spectrum of sample **138 (**binary mixture m-aramid (Nomex)/melamine) and of pure m-aramid (Nomex).

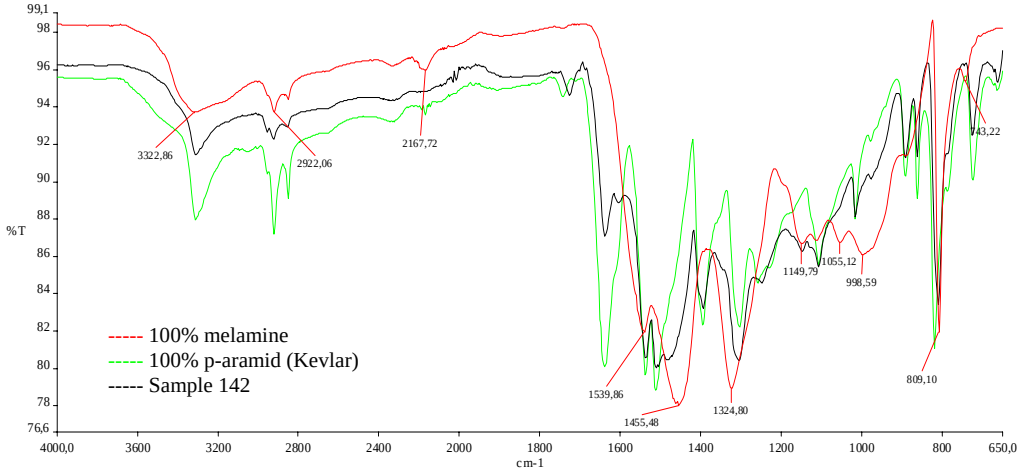


Fig.15: FT-IR spectrum of sample **142** (binary mixture p-aramid (Kevlar)/melamine) and of the pure component fibres.

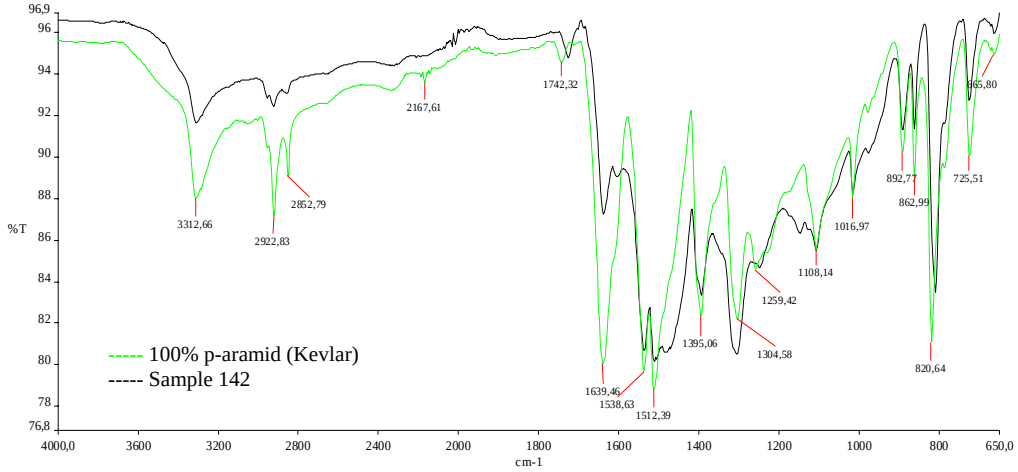


Fig.16: FT-IR spectrum of sample **142** (binary mixture p-aramid (Kevlar)/melamine) and of pure p-aramid (Kevlar).

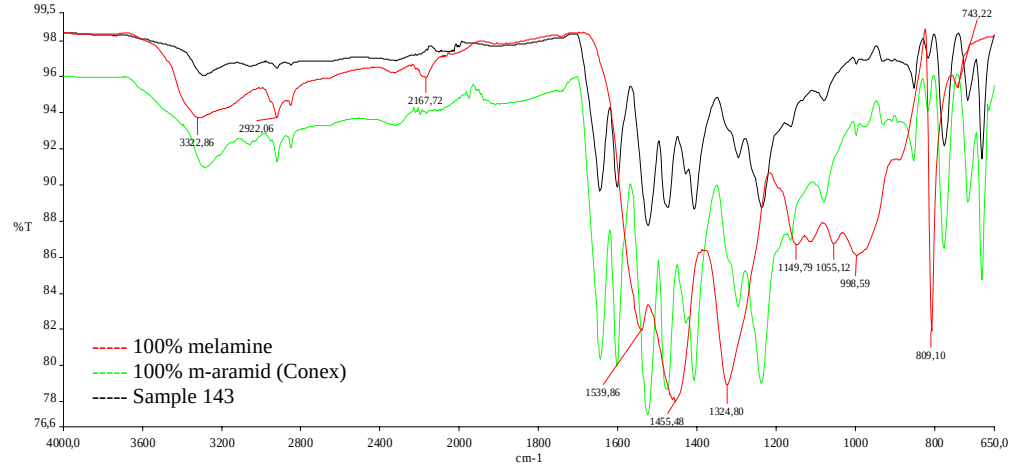


Fig.17: FT-IR spectrum of sample **143 (**binary mixture m-aramid (Conex)/melamine) and of the pure component fibres.

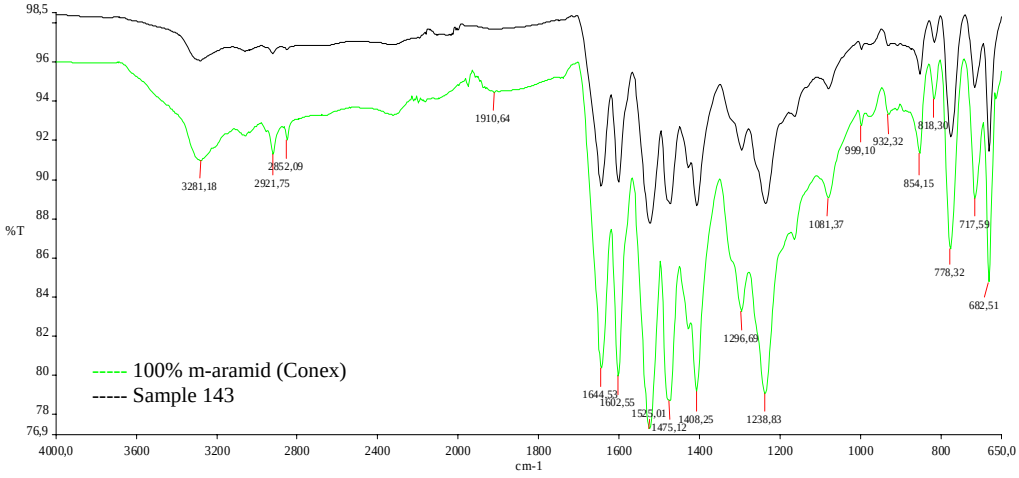


Fig.18: FT-IR spectrum of sample **143** (binary mixture m-aramid (Conex)/melamine) and of pure m-aramid (Conex).

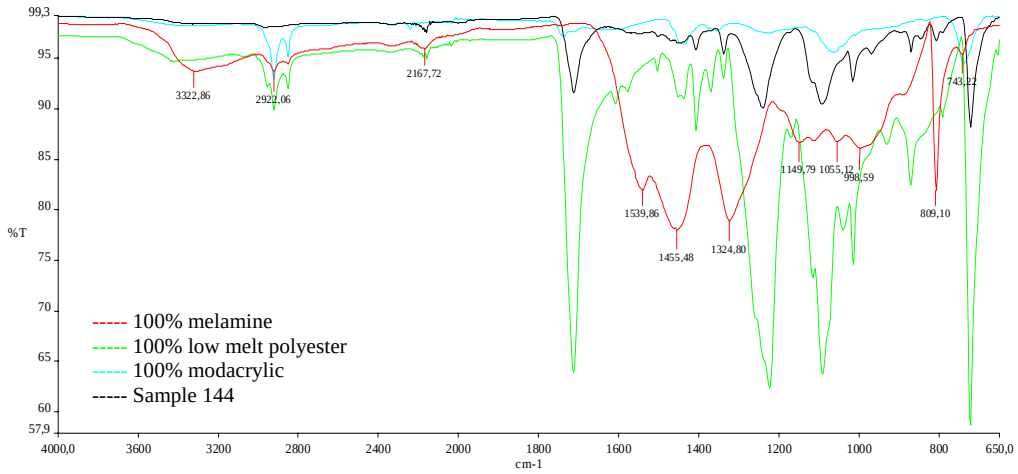


Fig.19: FT-IR spectrum of sample **144 (**ternary mixture melamine/polyester/modacrylic) and of the pure component fibres.

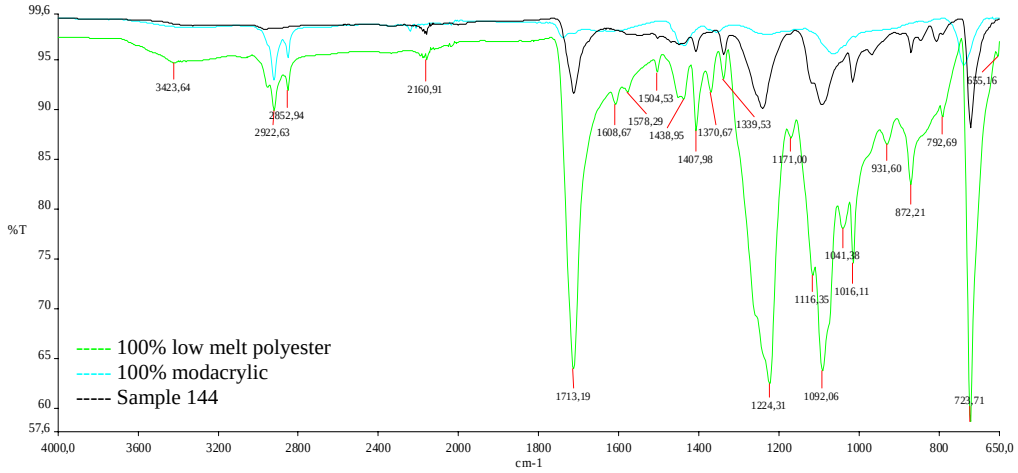


Fig.20: FT-IR spectrum of sample **144 (**ternary mixture melamine/polyester/modacrylic) and of pure polyester and modacrylic.

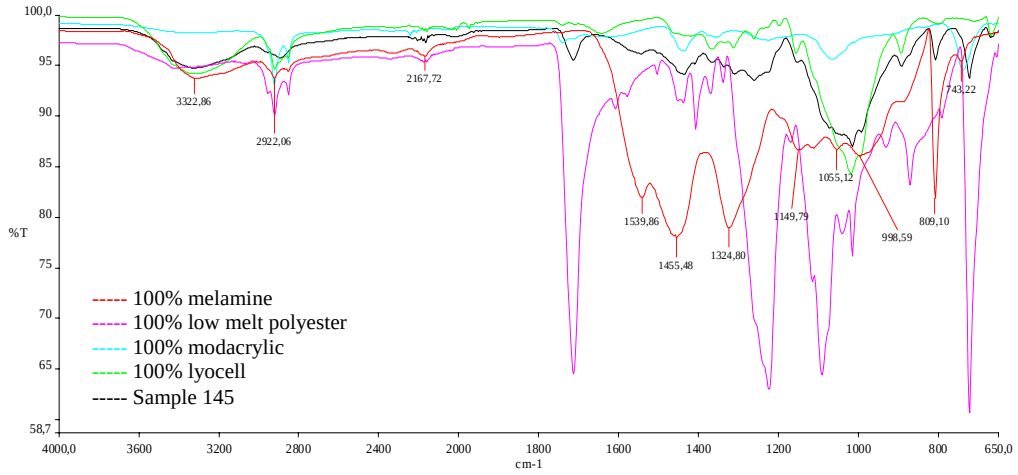


Fig.21: FT-IR spectrum of sample **145** (quaternary mixture melamine/polyester/modacrylic/lyocell) and of the pure component fibres.

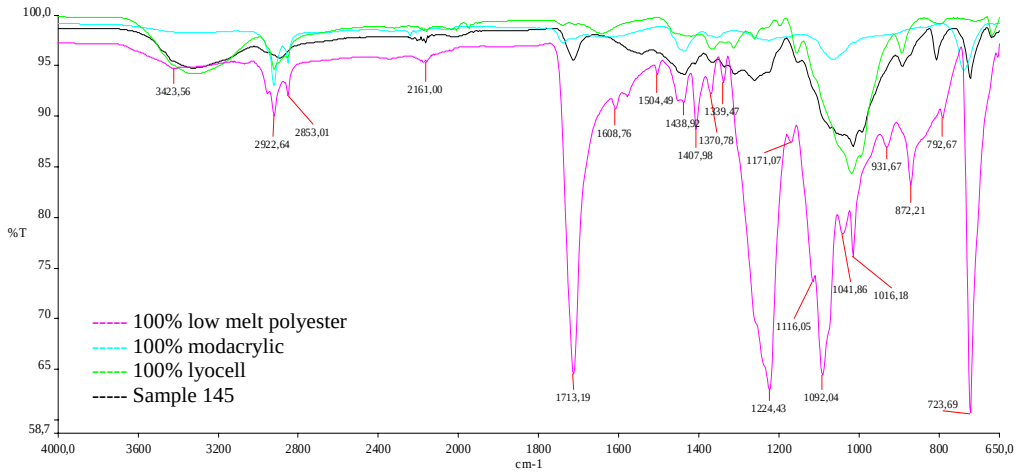


Fig.22: FT-IR spectrum of sample **145** (quaternary mixture melamine/polyester/modacrylic/lyocell) and of pure polyester, modacrylic and lyocell.

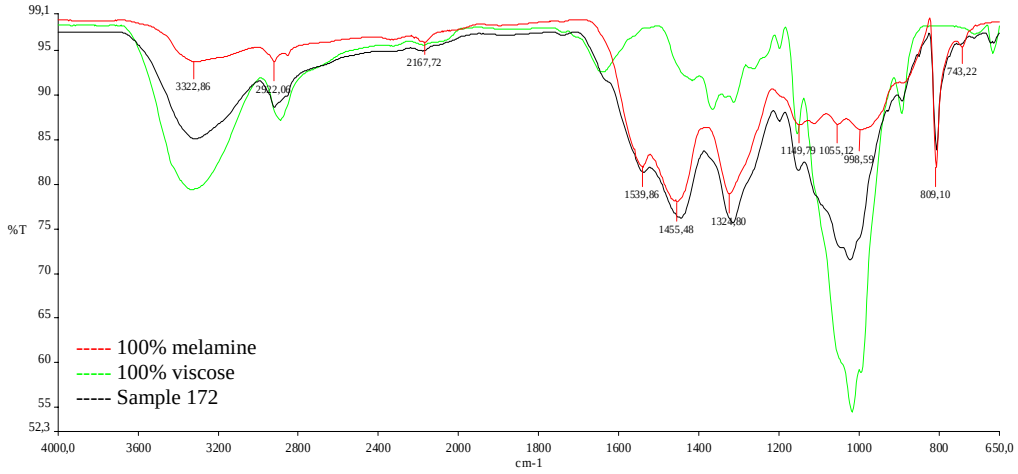


Fig.23: FT-IR spectrum of sample **172** (binary mixture melamine/viscose) and of the pure component fibres.

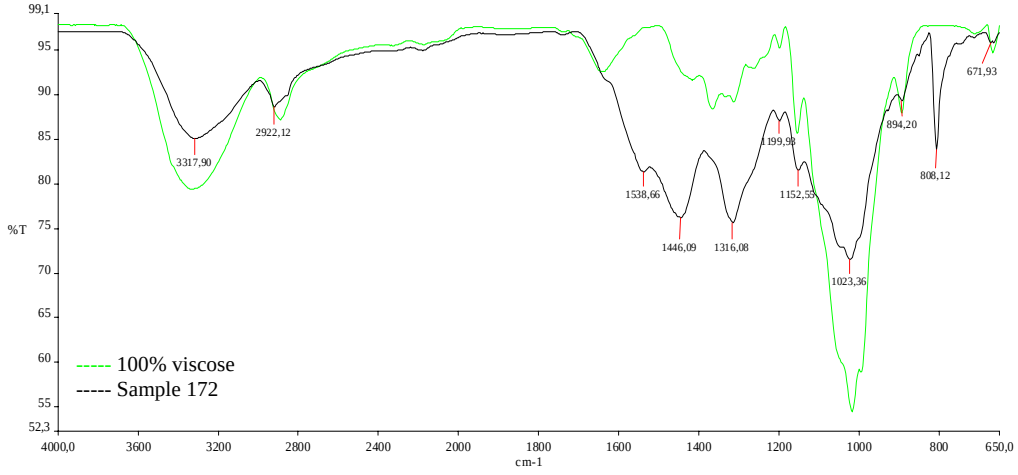


Fig.24: FT-IR spectrum of sample **172** (binary mixture melamine/viscose) and of pure viscose.

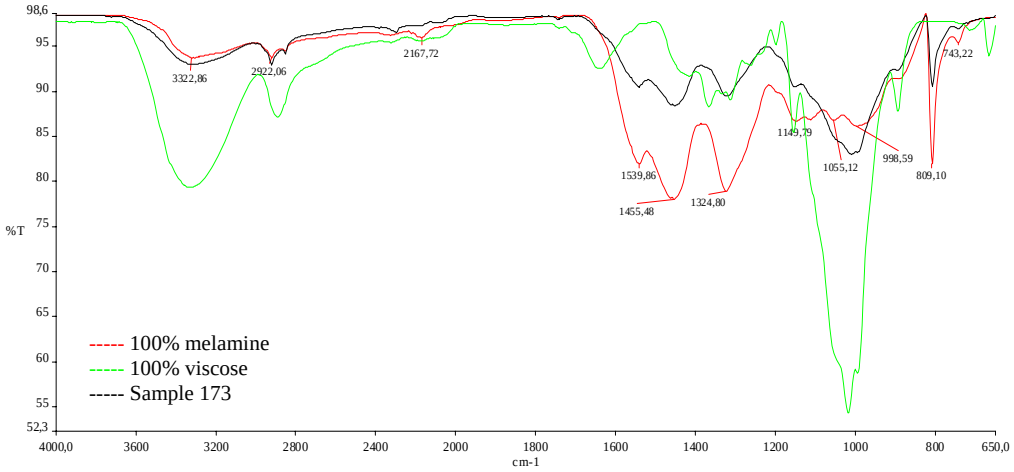


Fig.25: FT-IR spectrum of sample **173** (binary mixture melamine/Visil) and of the pure component fibres.

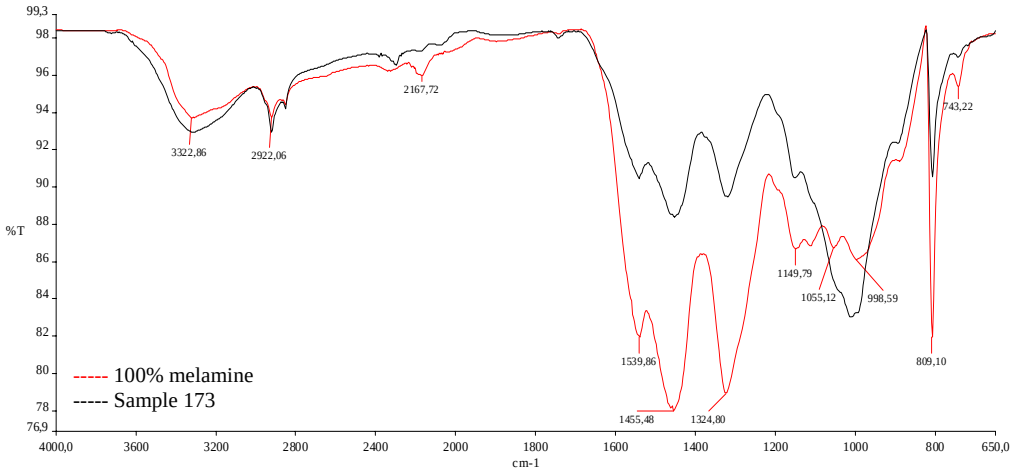


Fig.26: FT-IR spectrum of sample **173** (binary mixture melamine/Visil) and of pure melamine.

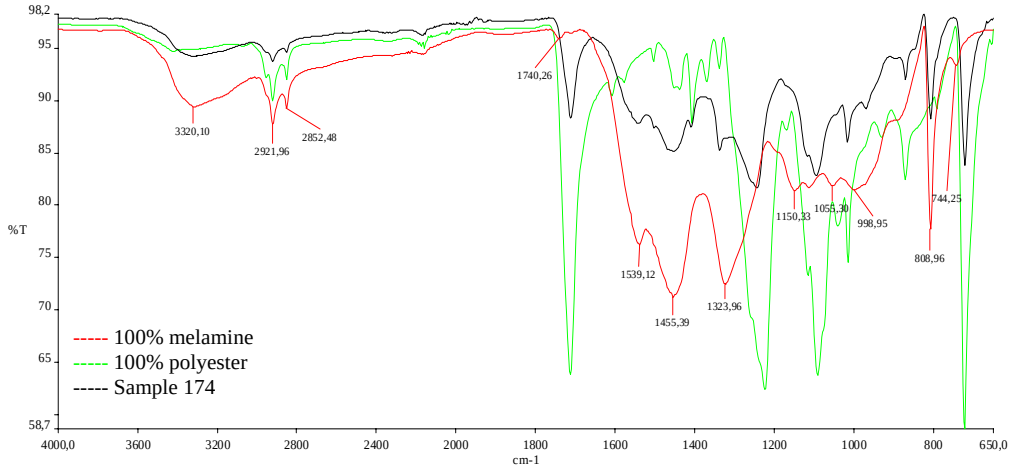


Fig.27: FT-IR spectrum of sample **174** (binary mixture melamine/polyester) and of the pure component fibres.

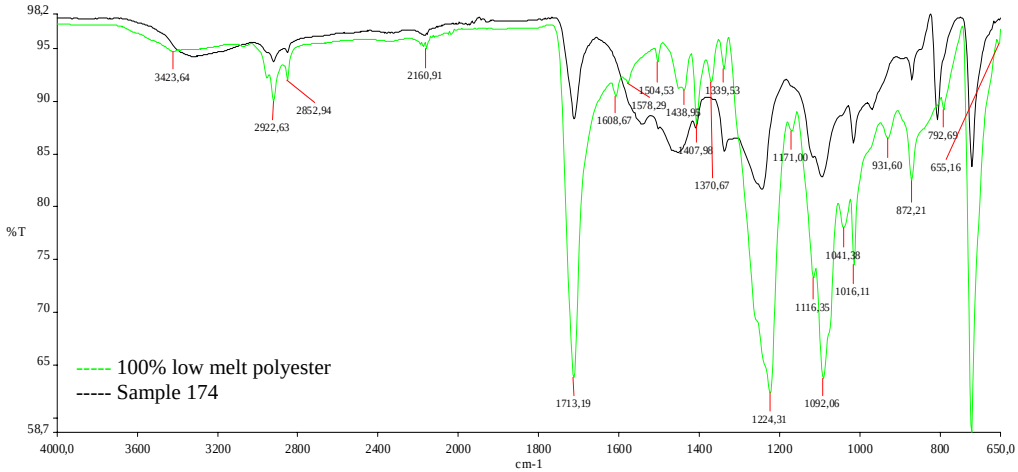


Fig.28: FT-IR spectrum of sample **174** (binary mixture melamine/polyester) and of pure polyester.

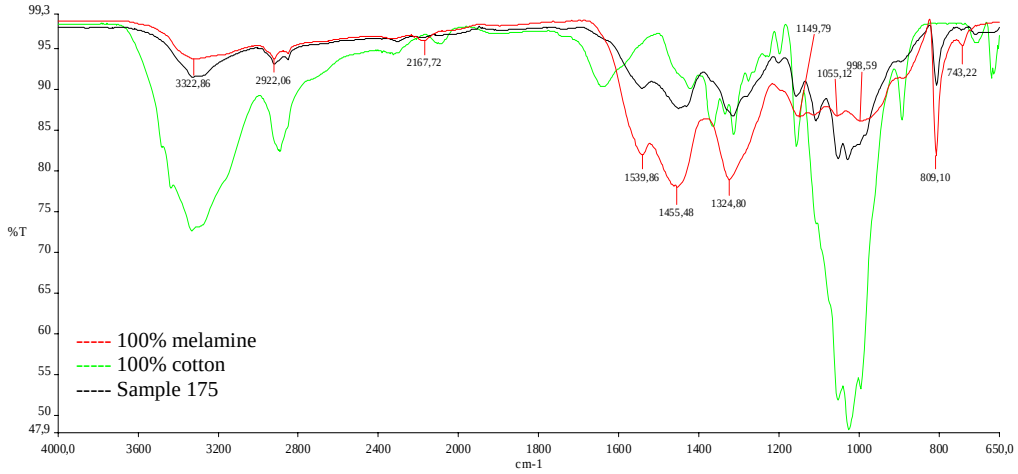


Fig.29: FT-IR spectrum of sample **175** (binary mixture melamine/cotton) and of the pure component fibres.

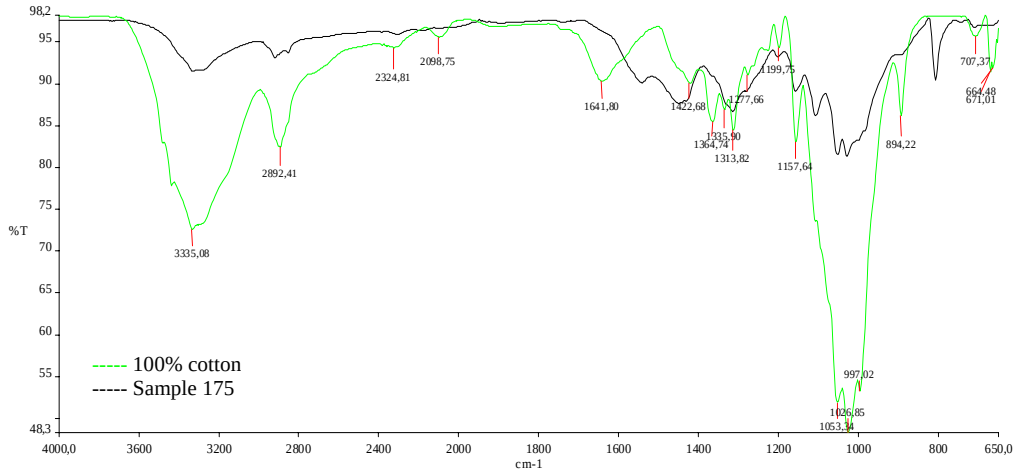


Fig.30: FT-IR spectrum of sample **175** (binary mixture melamine/cotton) and of pure cotton.

Annex IV

Thermo gravimetric Analysis

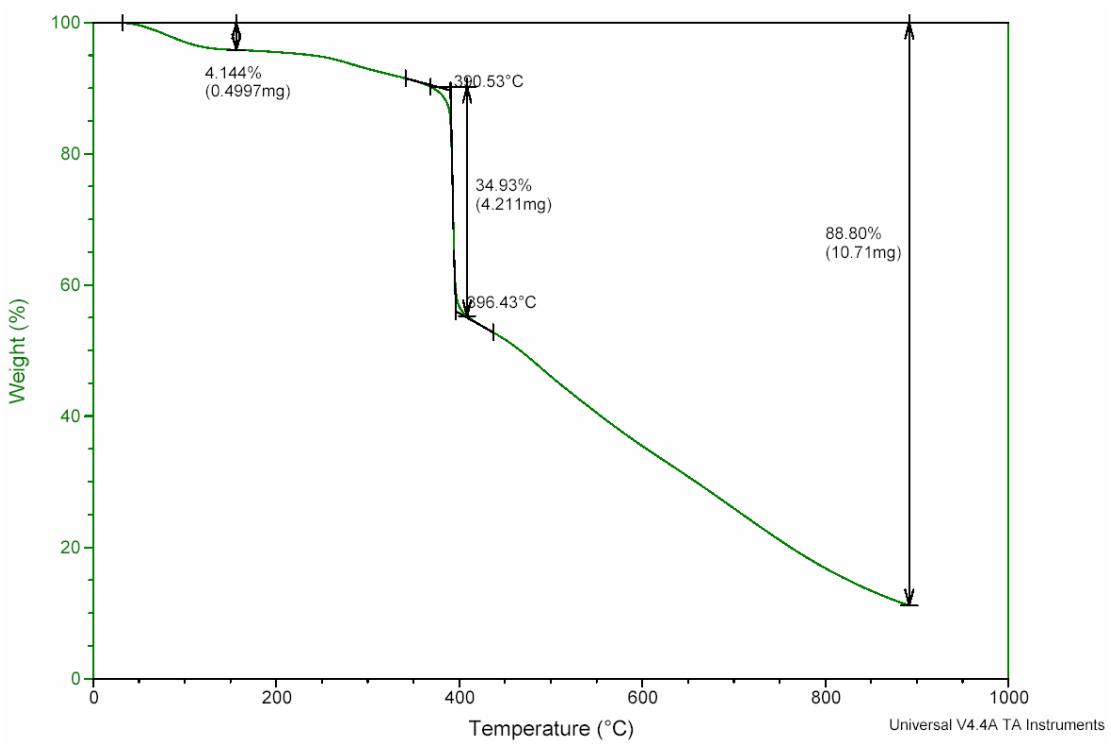


Fig.1: Thermo-gravimetric analysis of pure melamine (sample **136**).

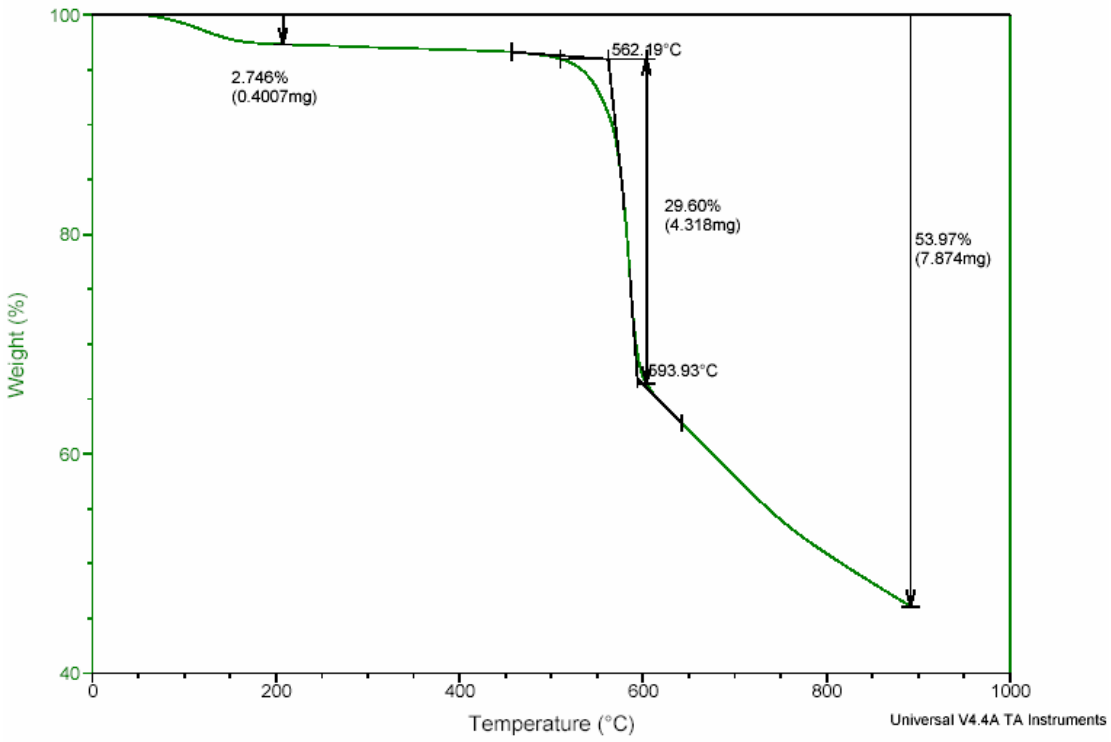


Fig.2: Thermo-gravimetric analysis of pure p-aramid (Kevlar) (sample **150**).

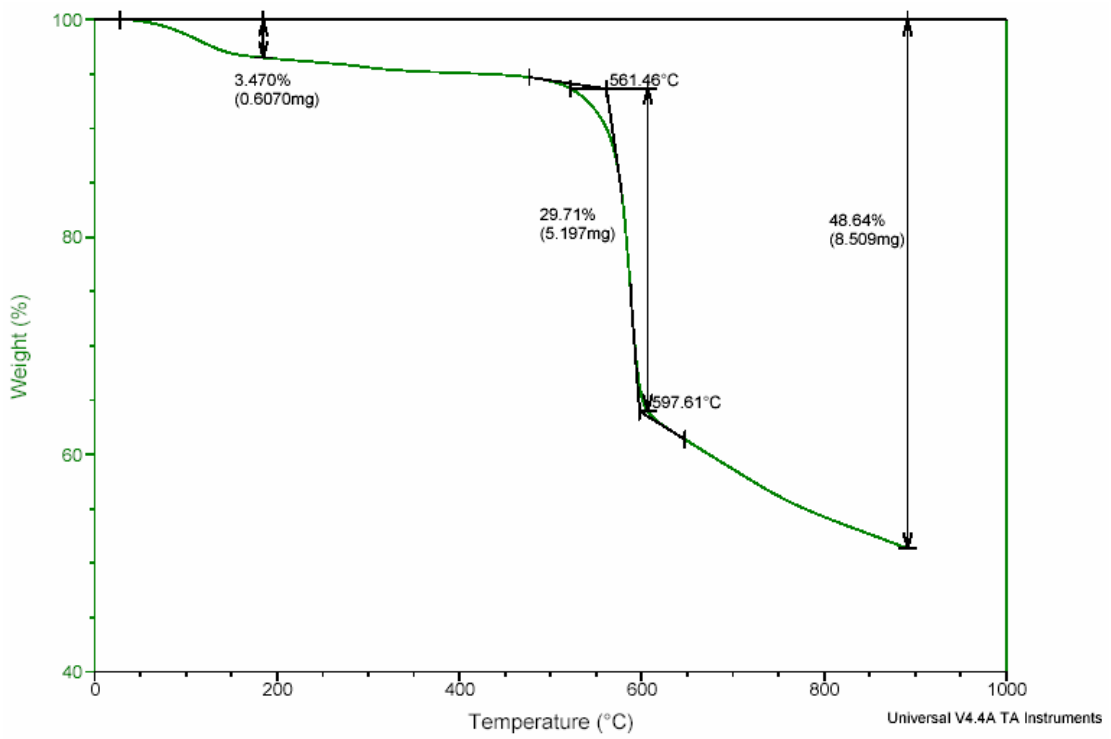


Fig.3: Thermo-gravimetric analysis of pure p-aramid (Twaron) (sample **182**).

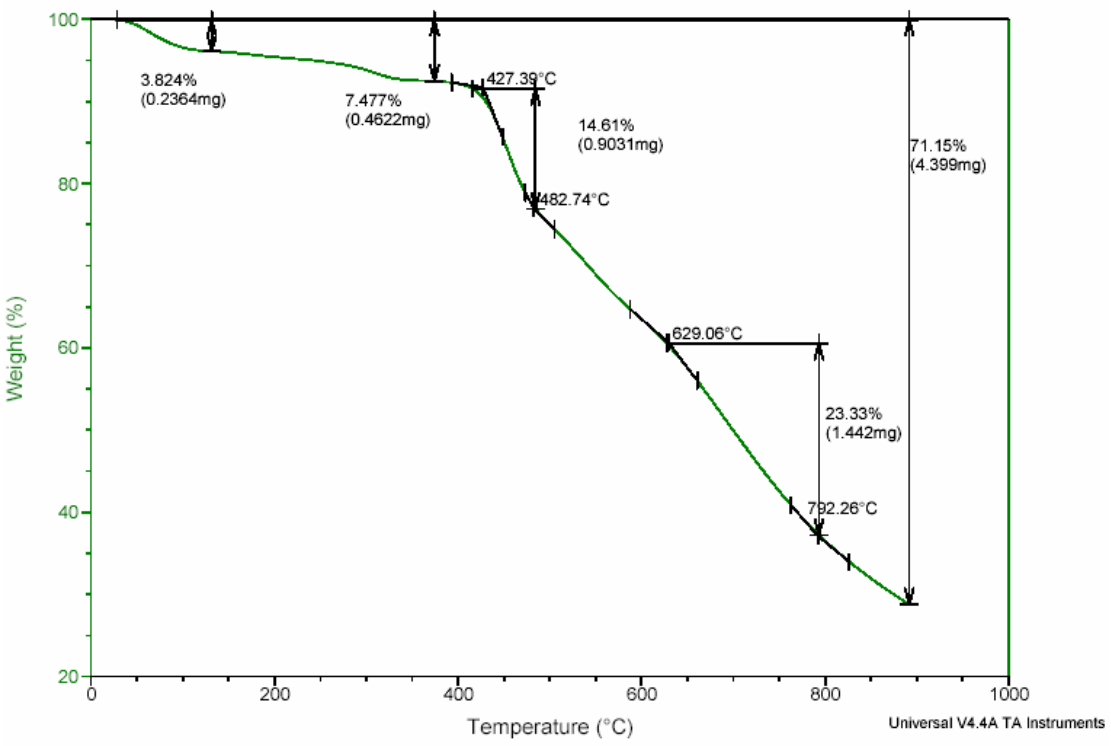


Fig.4: Thermo-gravimetric analysis of pure m-aramid (Nomex) (sample **151**).

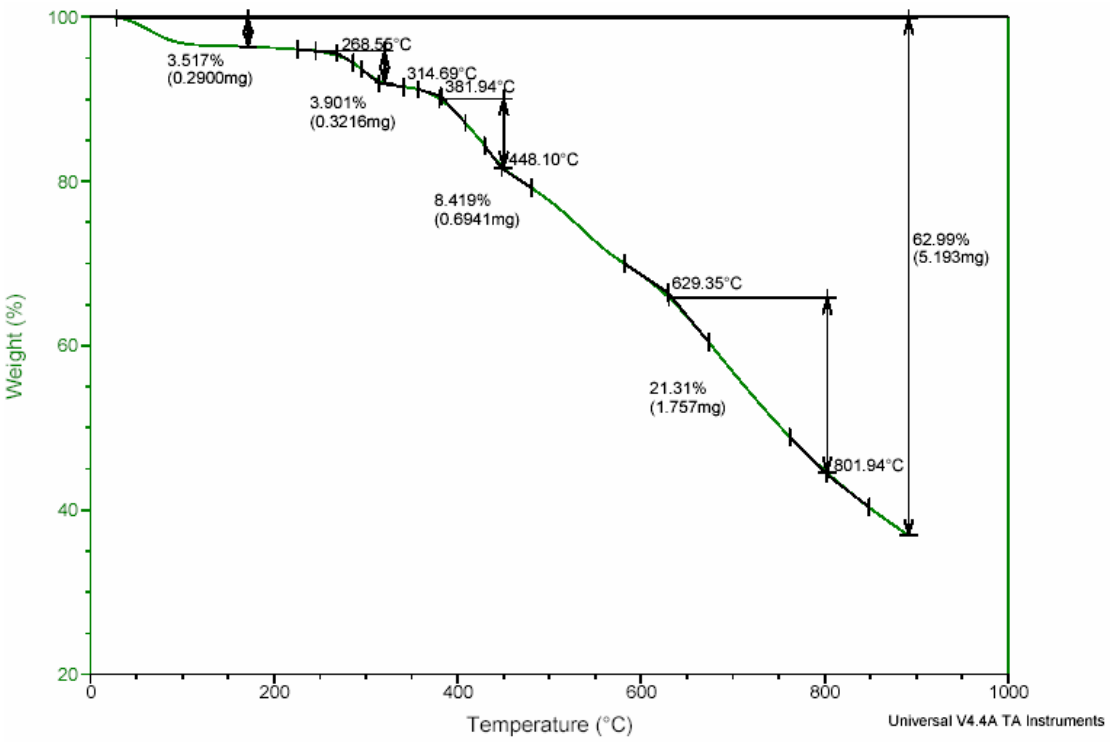


Fig.5: Thermo-gravimetric analysis of pure m-aramid (Conex) (sample **183**).

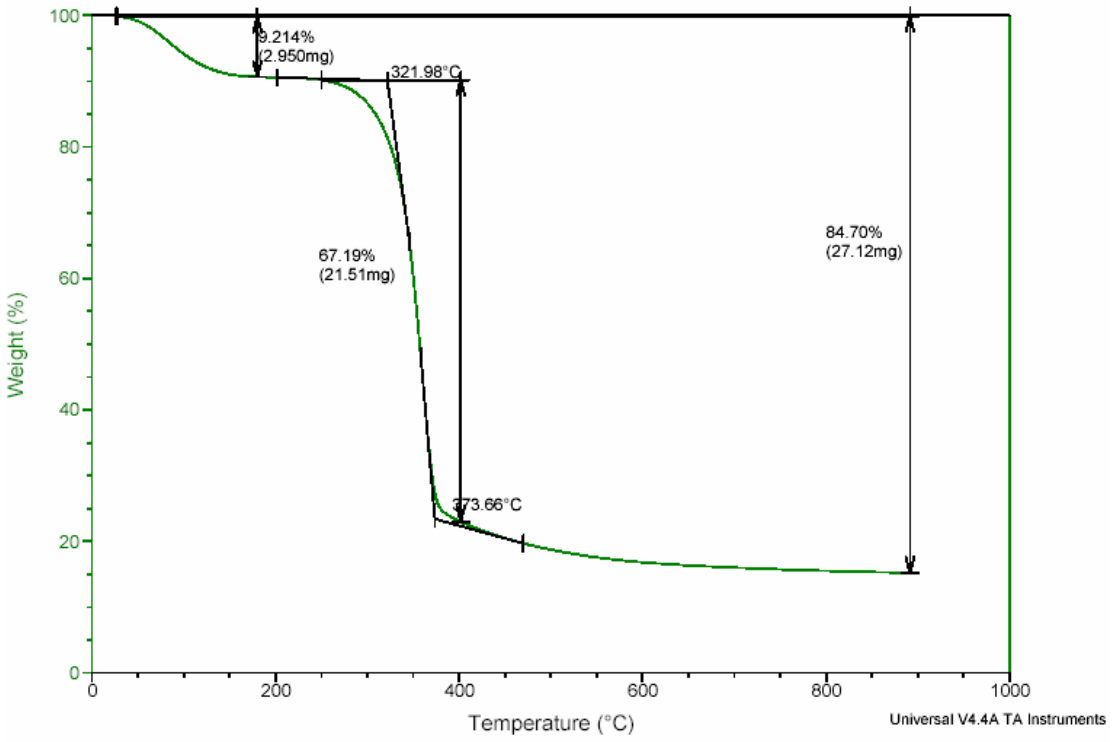


Fig.6: Thermo-gravimetric analysis of pure lyocell (sample **152**).

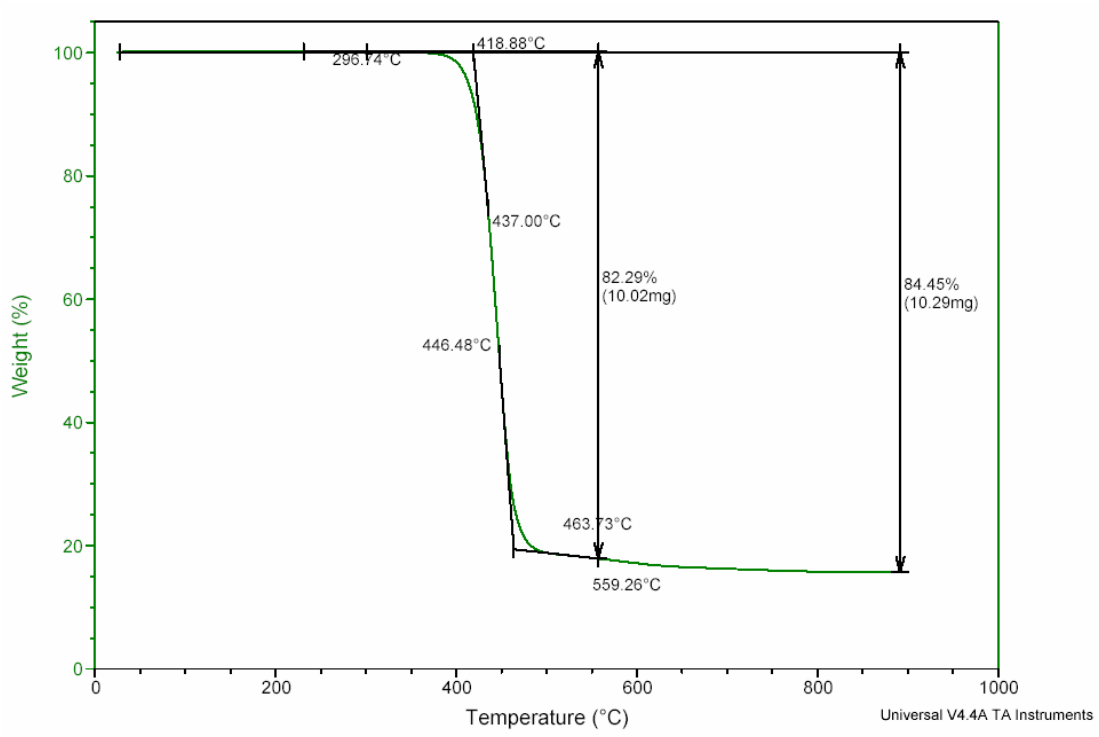


Fig.7: Thermo-gravimetric analysis of pure polyester (sample **153**).

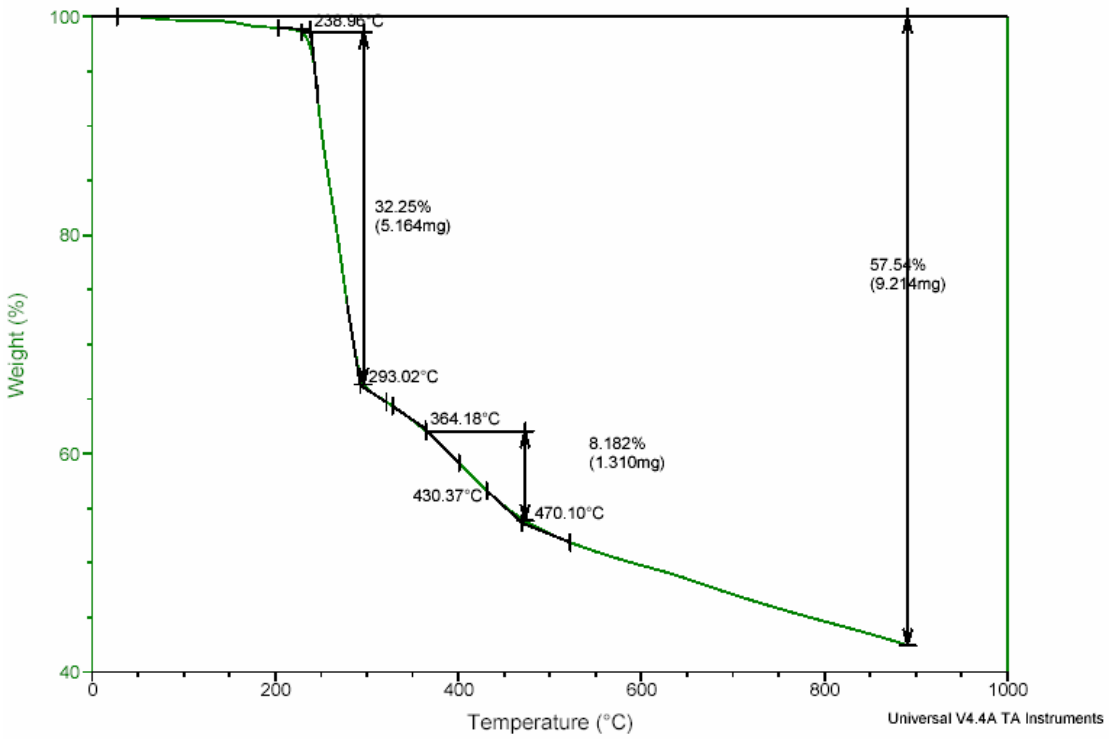


Fig.8: Thermo-gravimetric analysis of pure modacrylic (sample **154**).

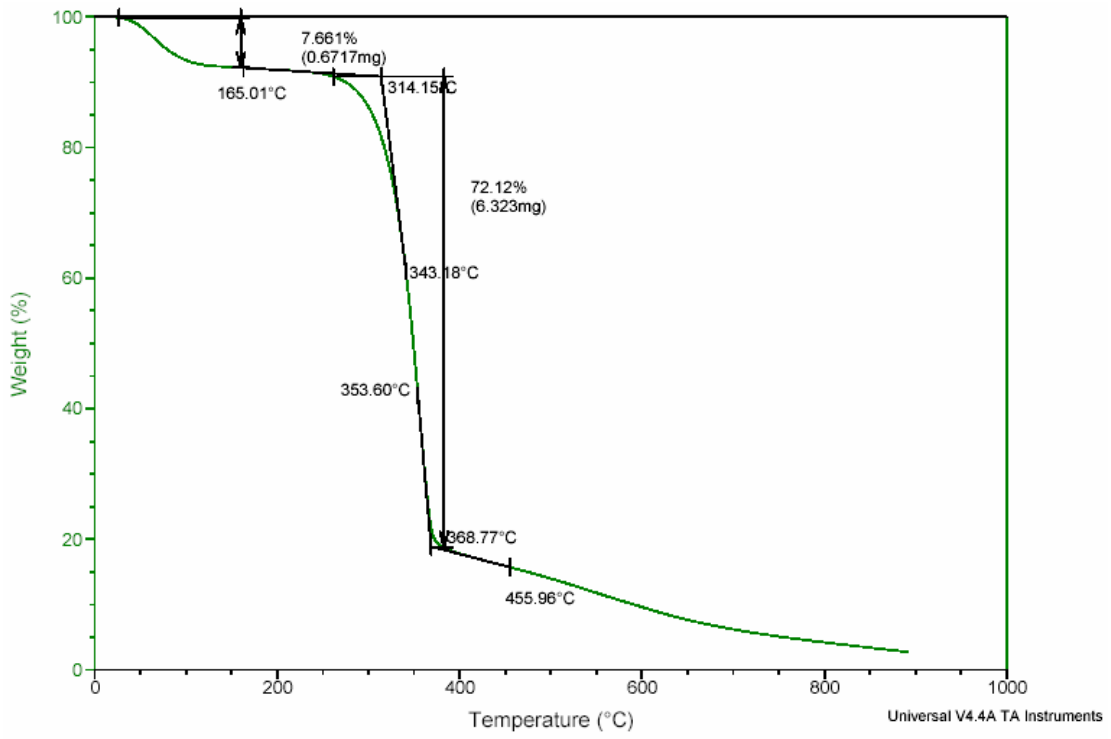


Fig.9: Thermo-gravimetric analysis of pure viscose (sample **034**).

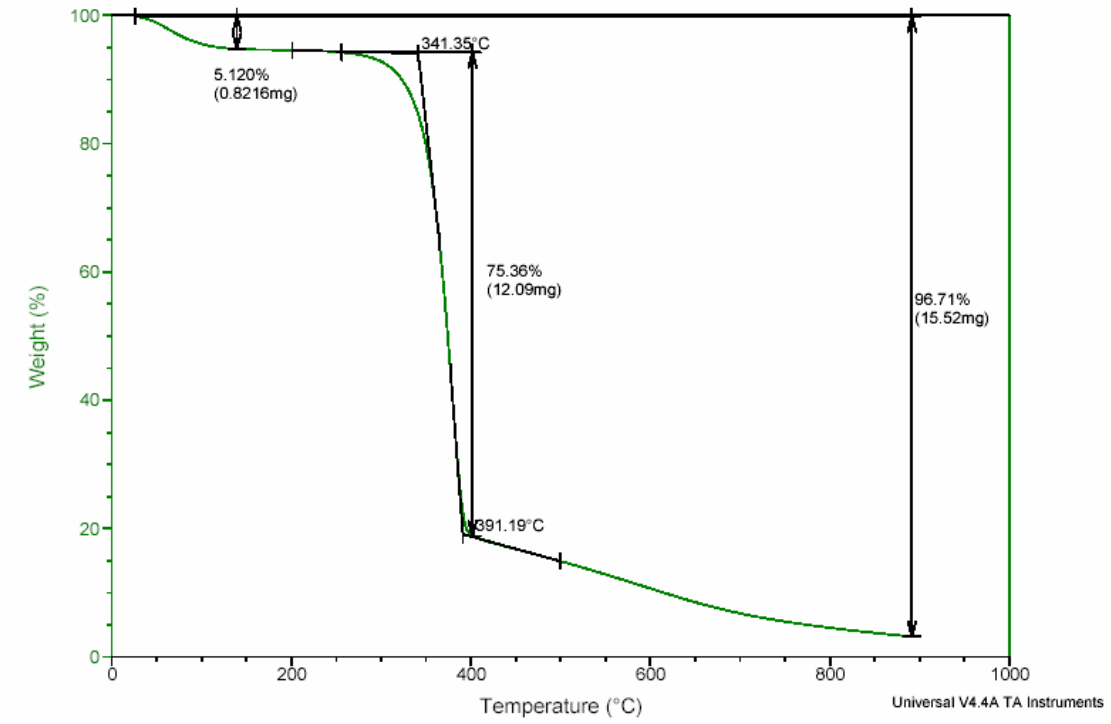


Fig.10: Thermo-gravimetric analysis of pure cotton (sample **005**).

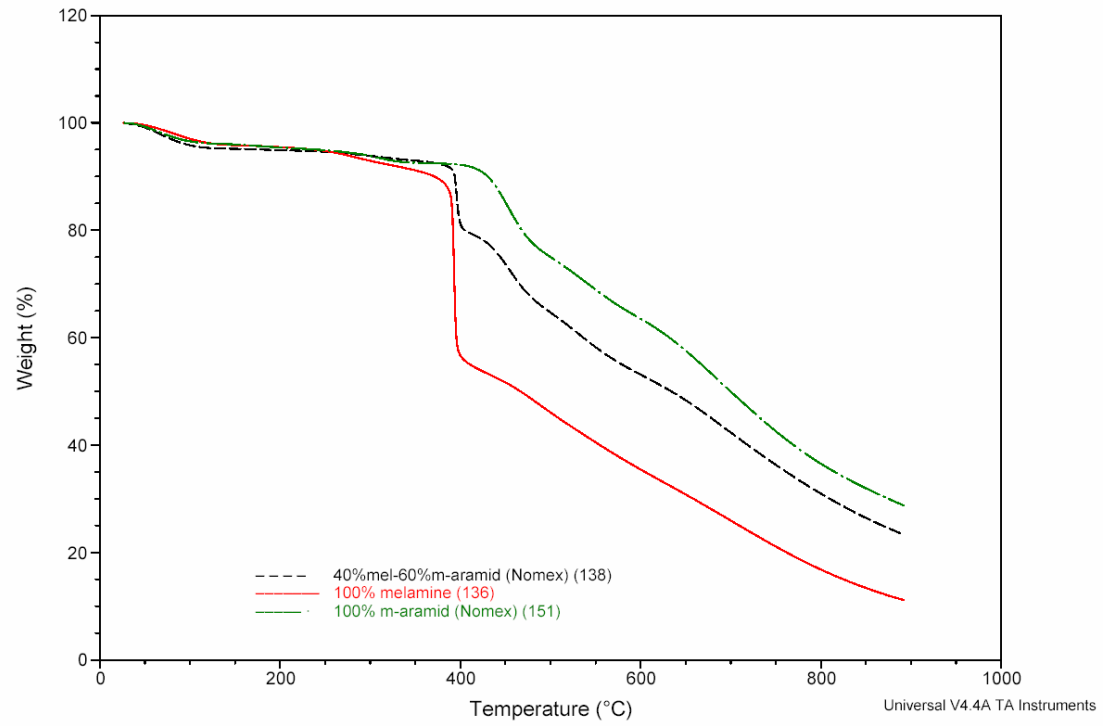


Fig.11: Thermo-gravimetric analysis of pure m-aramid (Nomex), melamine and one binary mixture of melamine with m-aramid (Nomex) (sample **138**).

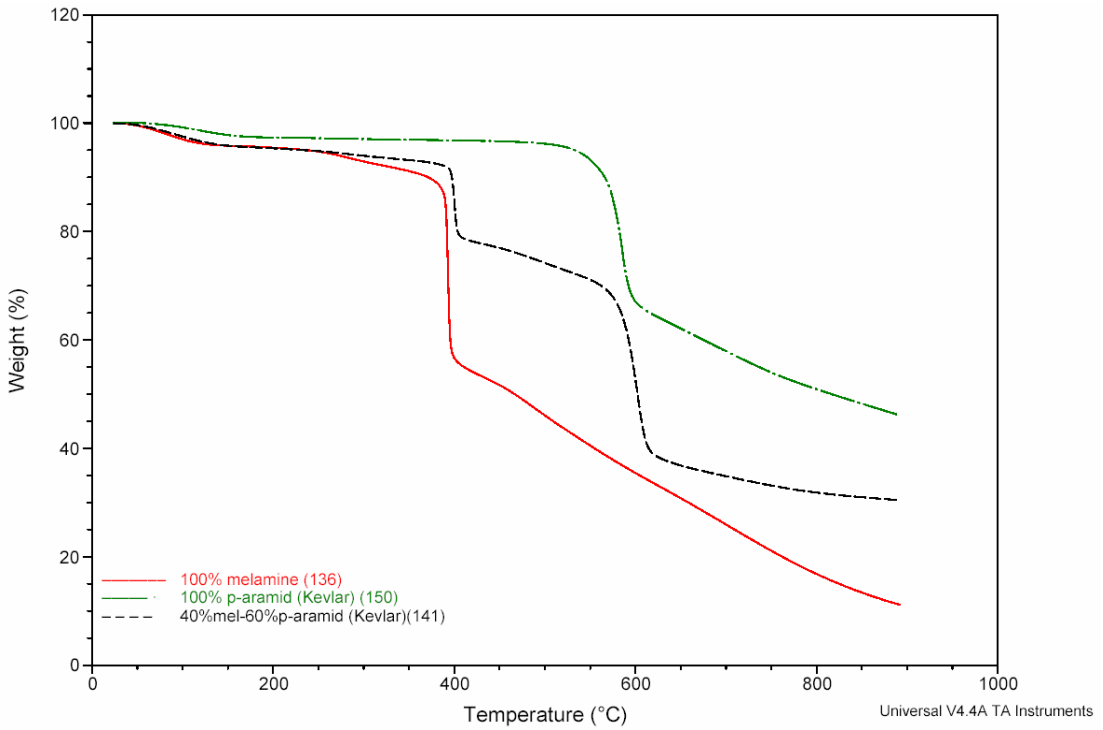


Fig.12: Thermo-gravimetric analysis of pure p-aramid (Kevlar), melamine and one binary mixture of melamine with p-aramid (Kevlar) (sample **141**).

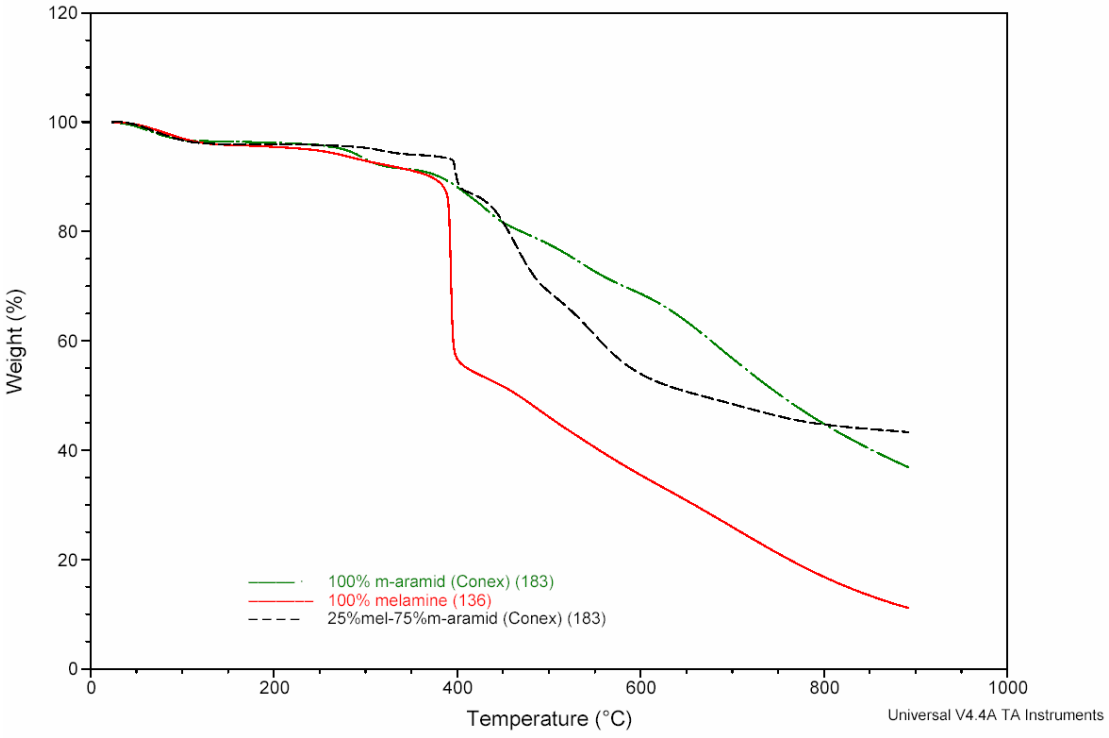


Fig.13: Thermo-gravimetric analysis of pure m-aramid (Conex), melamine and one binary mixture of melamine with m-aramid (Conex) (sample **143**).

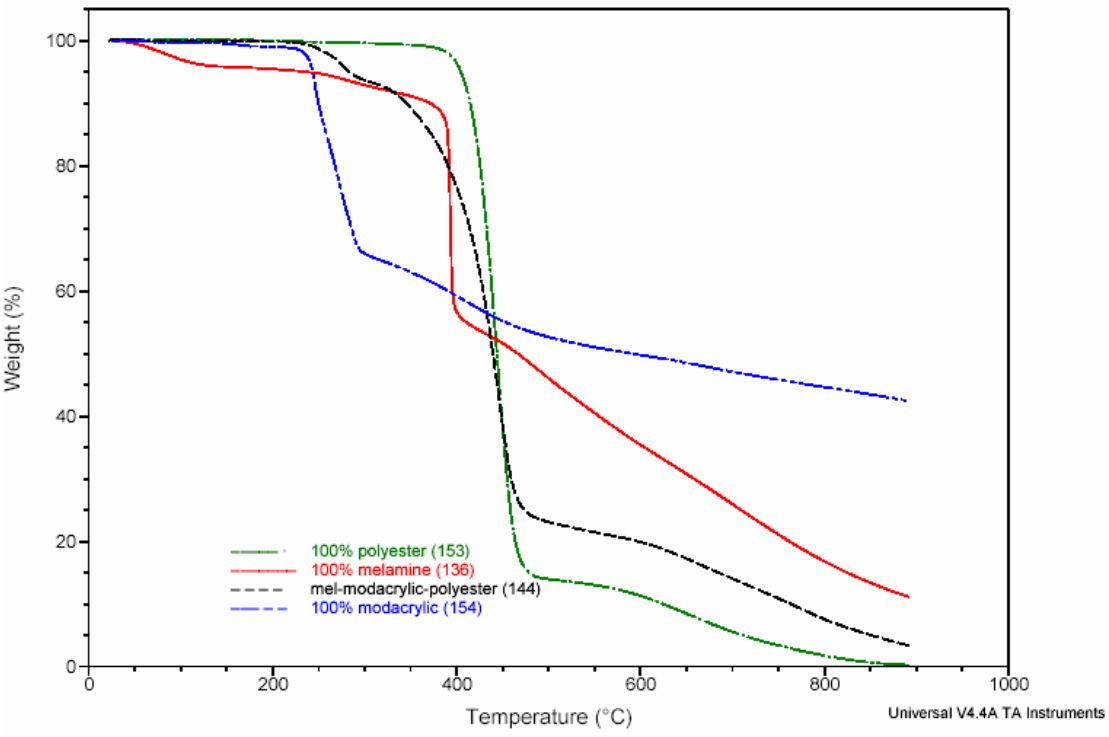


Fig.14: Thermo-gravimetric analysis of pure modacrylic, polyester, melamine and one ternary mixture of these fibres (sample **144**).

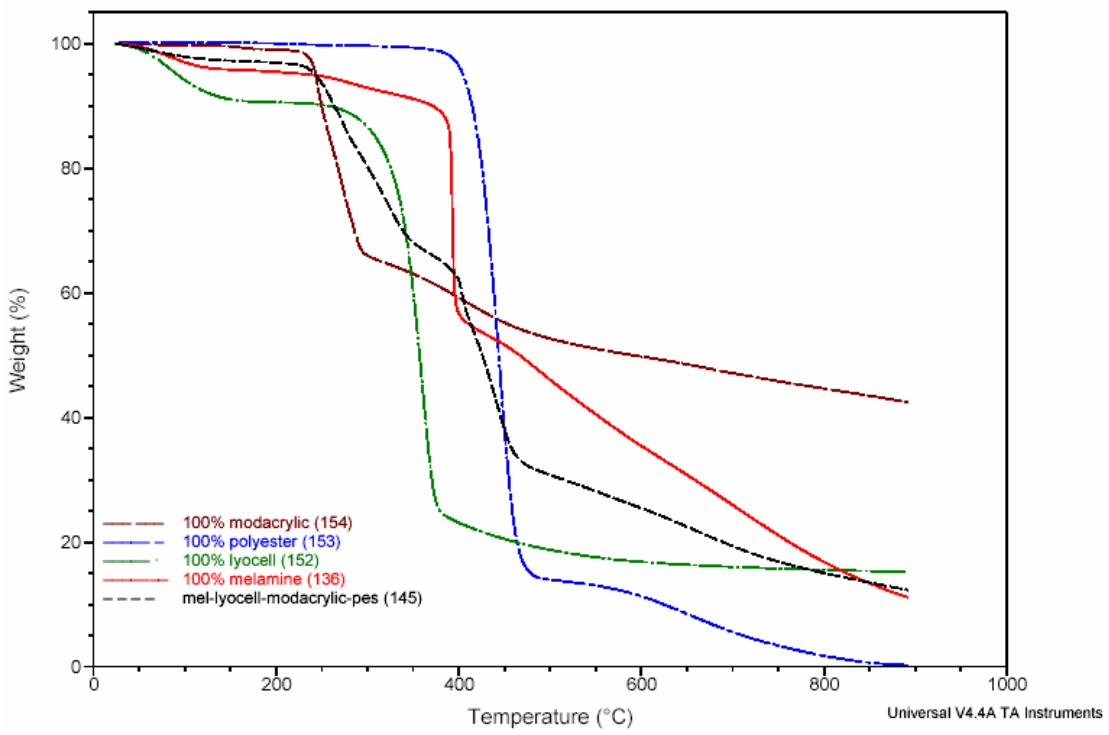


Fig.15: Thermo-gravimetric analysis of pure modacrylic, polyester, lyocell, melamine and one quaternary mixture of these fibers (sample **145**).

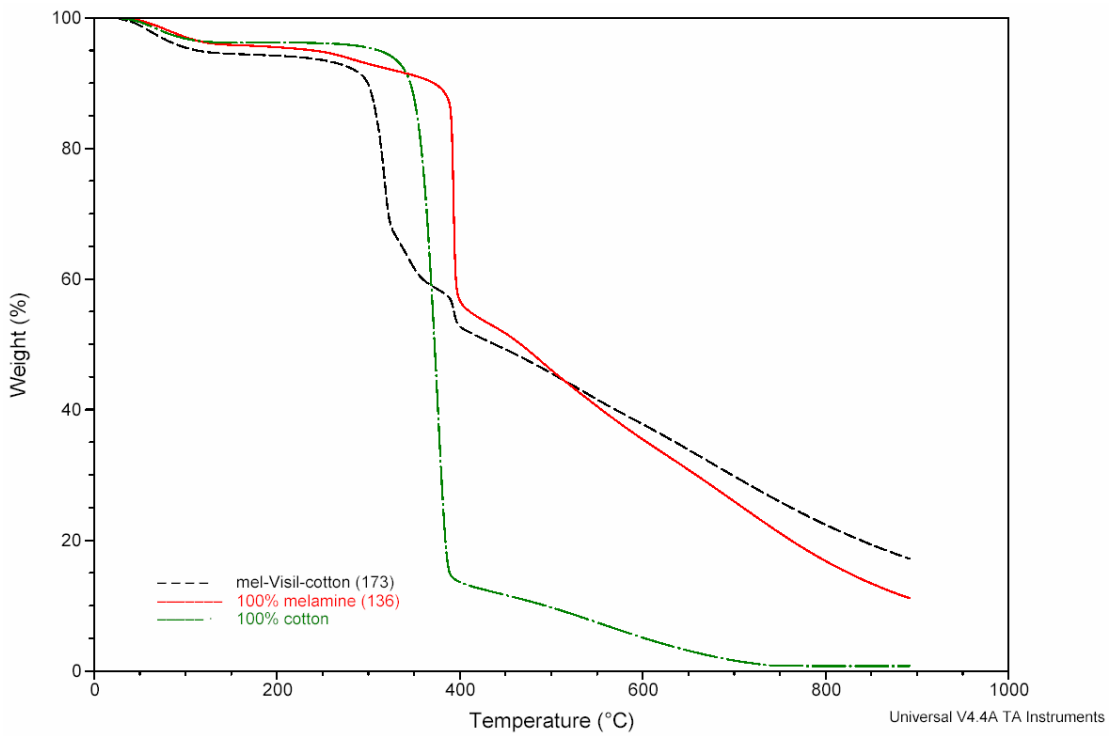


Fig.16: Thermo-gravimetric analysis of pure cotton, melamine and one binary mixture of melamine with Visil (sample **173**).

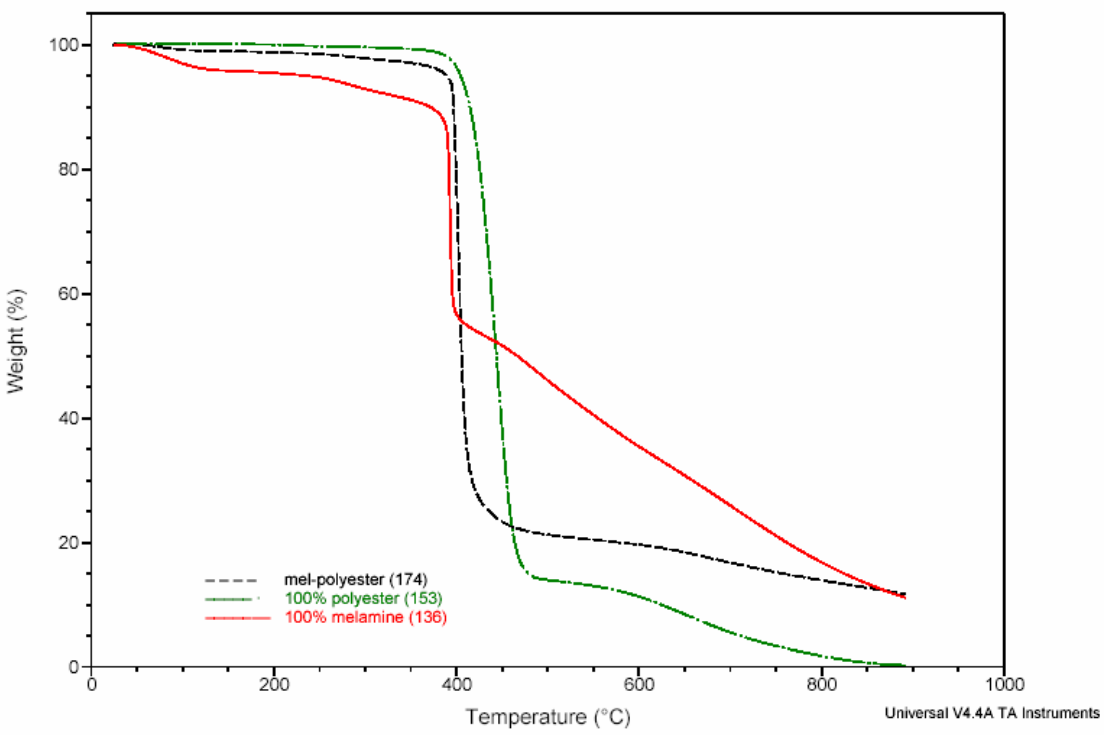


Fig.17: Thermo-gravimetric analysis of pure polyester, melamine and one binary mixture of melamine with polyester (sample **174**).

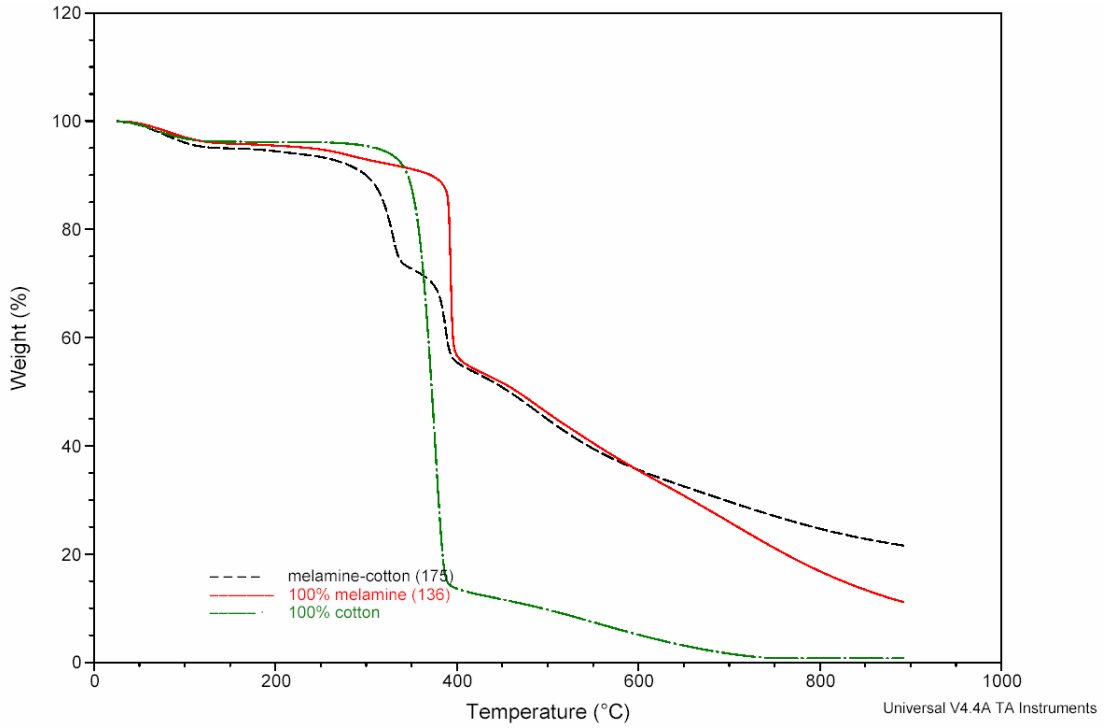


Fig.18: Thermo-gravimetric analysis of pure cotton, melamine and one binary mixture of melamine with cotton (sample **175**).

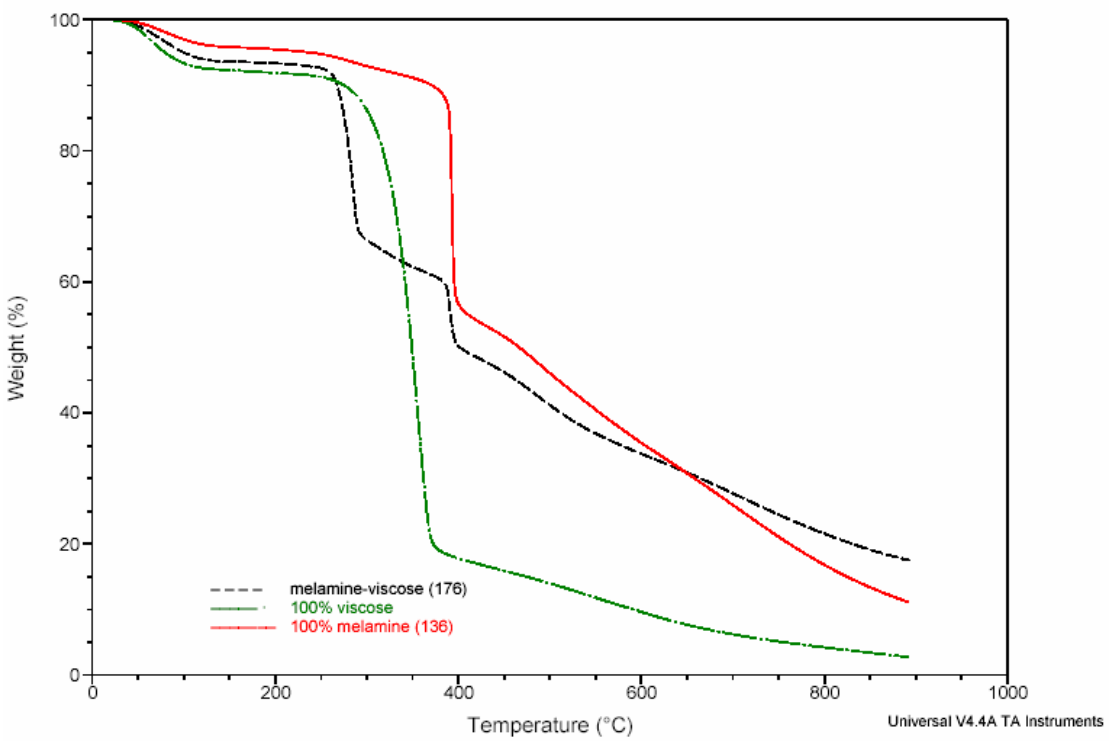


Fig.19: Thermo-gravimetric analysis of pure viscose, melamine and one binary mixture of melamine with viscose (sample **176**).

Annex V

Analysis of composition

Agreed Allowance (4 hours drying step)

100% melamine untreated (samples 136,137,171)

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
136-1	1.7605	1.8622	0.1017	5.78
136-2	1.9720	2.0862	0.1142	5.79
136-3	1.7036	1.8023	0.0987	5.79
136-4	1.6015	1.6939	0.0924	5.77
136-5	1.9048	2.0127	0.1079	5.66
136-6	1.9148	2.0236	0.1088	5.68
136-7	1.5169	1.6042	0.0873	5.76
136-8	1.5553	1.6451	0.0898	5.77
136-9	1.7271	1.8262	0.0991	5.74
136-10	1.7973	1.9011	0.1038	5.78
average				**5.75**
uncertainty				**0.03**
SD				**0.04**
RSD				**0.78**

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
137-1	1.8316	1.9512	0.1196	6.53
137-2	1.7105	1.8217	0.1112	6.50
137-3	1.9822	2.1112	0.1290	6.51
137-4	1.7703	1.8839	0.1136	6.42
137-5	1.6875	1.7977	0.1102	6.53
137-6	1.8432	1.9633	0.1201	6.52
137-7	2.049	2.1834	0.1344	6.56
137-8	1.8185	1.9359	0.1174	6.46
137-9	1.8432	1.9641	0.1209	6.56
137-10	1.7614	1.8766	0.1152	6.54
average				**6.51**
uncertainty				**0.03**
SD				**0.05**
RSD				**0.69**

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
171-1	1.0517	1.1182	0.0665	6.32
171-2	0.9860	1.0482	0.0622	6.31
171-3	1.0353	1.1005	0.0652	6.30
171-4	0.9391	0.9990	0.0599	6.38
171-5	0.9901	1.0518	0.0617	6.23
171-6	1.039	1.1034	0.0644	6.20
171-7	1.005	1.0683	0.0633	6.30
171-8	1.0047	1.0675	0.0628	6.25
171-9	0.9954	1.0573	0.0619	6.22
171-10	0.993	1.0545	0.0615	6.19
average				**6.27**
uncertainty				**0.04**
SD				**0.06**
RSD				**0.97**

100% melamine pre-treated (samples 136,137,171)

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
136-1	1.7524	1.8830	0.1306	7.45
136-2	1.7390	1.8660	0.1270	7.30
136-3	1.7293	1.8573	0.1280	7.40
136-4	1.7602	1.8886	0.1284	7.29
136-5	1.6604	1.7803	0.1199	7.22
136-6	1.6782	1.8028	0.1246	7.42
136-7	1.7896	1.9216	0.1320	7.38
136-8	1.8591	1.9939	0.1348	7.25
136-9	1.7653	1.8953	0.1300	7.36
136-10	1.8704	2.0079	0.1375	7.35
average				7.34
uncertainty				0.05
SD				0.08
RSD				1.03

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
137-1	2.0384	2.1931	0.1547	7.59
137-2	1.3195	1.4192	0.0997	7.56
137-3	1.5585	1.6757	0.1172	7.52
137-4	1.3464	1.4503	0.1039	7.72
137-5	1.8009	1.9378	0.1369	7.60
137-6	1.8192	1.9561	0.1369	7.53
137-7	1.5592	1.6760	0.1168	7.49
137-8	2.1434	2.3068	0.1634	7.62
137-9	2.2491	2.4217	0.1726	7.67
137-10	2.1609	2.3252	0.1643	7.60
average				7.59
uncertainty				0.05
SD				0.07
RSD				0.93

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
171-1	1.0532	1.1292	0.0760	7.22
171-2	0.9159	0.9829	0.0670	7.32
171-3	1.0425	1.1182	0.0757	7.26
171-4	0.9972	1.0698	0.0726	7.28
171-5	1.0889	1.1681	0.0792	7.27
171-6	1.0227	1.0975	0.0748	7.31
171-7	1.0055	1.0790	0.0735	7.31
171-8	0.9777	1.0493	0.0716	7.32
171-9	1.0022	1.0751	0.0729	7.27
171-10	1.0035	1.0764	0.0729	7.26
average				7.28
uncertainty				0.02
SD				0.03
RSD				0.45

Agreed Allowance (16 hours drying step)

100% melamine untreated (samples 136,137,179)

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
136-1	1.6361	1.7419	0.1058	6.47
136-2	1.5617	1.6645	0.1028	6.58
136-3	1.7898	1.9066	0.1168	6.53
136-4	1.659	1.7662	0.1072	6.46
136-5	1.7951	1.9124	0.1173	6.53
179-6	0.9158	0.9757	0.0599	6.54
179-7	0.8964	0.9554	0.0590	6.58
179-8	1.108	1.1801	0.0721	6.51
179-9	0.9813	1.0454	0.0641	6.53
179-10	0.8837	0.9413	0.0576	6.52
average				**6.53**
uncertainty				**0.03**
SD				**0.04**
RSD				**0.62**

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
137-1	1.0726	1.1465	0.0739	6.89
137-2	1.1498	1.2276	0.0778	6.77
137-3	1.2326	1.3172	0.0846	6.86
137-4	1.4552	1.5558	0.1006	6.91
137-5	1.5567	1.6637	0.1070	6.87
137-6	1.1176	1.1949	0.0773	6.92
137-7	1.0843	1.1601	0.0758	6.99
137-8	0.9934	1.0628	0.0694	6.99
137-9	1.0321	1.1038	0.0717	6.95
137-10	0.9938	1.0637	0.0699	7.03
average				**6.92**
uncertainty				**0.05**
SD				**0.08**
RSD				**1.11**

100% melamine pre-treated (samples 136,137,179)

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
136-1	0.9642	1.0330	0.0688	7.14
136-2	1.2656	1.3562	0.0906	7.16
136-3	0.8044	0.8618	0.0574	7.14
136-4	1.3206	1.4154	0.0948	7.18
136-5	0.9140	0.9792	0.0652	7.13
179-6	1.1255	1.1993	0.0738	6.56
179-7	1.2381	1.3194	0.0813	6.57
179-8	1.1855	1.2645	0.0790	6.66
179-9	1.3248	1.4114	0.0866	6.54
179-10	1.076	1.1471	0.0711	6.61
average				**6.87**
uncertainty				**0.21**
SD				**0.30**
RSD				**4.34**

JRC code	dried sample mass	wet sample mass	water weight	agreed allowance
	g	g	g	%
137-1	1.0839	1.1617	0.0778	7.18
137-2	1.1830	1.2689	0.0859	7.26
137-3	1.2426	1.3327	0.0901	7.25
137-4	1.2494	1.3399	0.0905	7.24
137-5	1.2260	1.3154	0.0894	7.29
137-6	1.3734	1.4716	0.0982	7.15
137-7	2.0074	2.1528	0.1454	7.24
137-8	1.2852	1.3768	0.0916	7.13
137-9	1.3515	1.4487	0.0972	7.19
137-10	1.5053	1.6137	0.1084	7.20
average				**7.21**
uncertainty				**0.04**
SD				**0.05**
RSD				**0.73**

Pre-treatment with petroleum ether in Soxhlet

100 % melamine (samples 136,137)

JRC code	sample mass	melamine mass	melamine mass loss	melamine mass loss
	g	g	g	%
136-1	1.0761	1.0700	0.0061	0.58
136-2	1.0278	1.0225	0.0053	0.52
136-3	0.9774	0.9704	0.0070	0.72
136-4	1.0646	1.0788	0.0058	0.54
136-5	1.0331	1.0270	0.0061	0.59
136-6	0.9860	0.9785	0.0075	0.76
136-7	1.0600	1.0727	0.0073	0.69
136-8	1.0051	0.9981	0.0070	0.70
136-9	1.0069	0.9990	0.0079	0.78
136-10	1.0123	1.0051	0.0072	0.71
136-11	3.2362	3.2090	0.0272	0.84
136-12	3.1382	3.1156	0.0226	0.72
136-13	3.1008	3.0753	0.0255	0.82
136-14	3.4189	3.3917	0.0272	0.80
136-15	2.8678	2.8491	0.0187	0.65
136-16	3.4182	3.3892	0.0290	0.85
average			**0.0136**	**0.70**
uncertainty			**0.0050**	**0.06**
SD			**0.0094**	**0.10**
RSD			**69.44**	**14.88**

JRC code	sample mass	melamine mass	melamine mass loss	melamine mass loss
	g	g	g	%
137-1	1.2542	1.2431	0.0111	0.89
137-2	1.1793	1.1692	0.0101	0.86
137-3	0.9902	0.9827	0.0075	0.76
137-4	1.0443	1.0363	0.0080	0.77
137-5	1.0407	1.0345	0.0062	0.60
137-6	1.0496	1.0417	0.0079	0.75
137-7	1.0141	1.0052	0.0089	0.88
137-8	0.9698	0.9627	0.0071	0.73
137-9	1.0086	1.0015	0.0071	0.70
137-10	1.0221	1.0166	0.0055	0.54
average			**0.0079**	**0.75**
uncertainty			**0.0012**	**0.08**
SD			**0.0017**	**0.11**
RSD			**21.47**	**15.29**

40% melamine-60% m-aramid (Nomex) (sample 138)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
138-1	5.8447	5.7977	0.0470	0.80
138-2	5.7149	5.6647	0.0502	0.88
138-3	5.4992	5.4560	0.0432	0.79
average			0.0468	0.82
uncertainty			0.0087	0.12
SD			0.0035	0.05
RSD			7.49	5.97

40% melamine-60% m-aramid (Nomex) (sample 139)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
139-1	4.8006	4.7654	0.0352	0.73
139-2	6.1387	6.0915	0.0472	0.77
139-3	5.1677	5.1319	0.0358	0.69
average			0.0394	0.73
uncertainty			0.0168	0.09
SD			0.0068	0.04
RSD			17.16	5.21

50% melamine-50% m-aramid (Nomex) (sample 140)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
140-1	4.3523	4.3419	0.0104	0.24
140-2	3.3229	3.3145	0.0084	0.25
140-3	4.2344	4.2237	0.0107	0.25
140-4	3.7926	3.7836	0.0090	0.24
140-5	2.8790	2.8717	0.0073	0.25
average			0.0092	0.25
uncertainty			0.0018	0.01
SD			0.0014	0.01
RSD			15.41	3.31

40% melamine-60% p-aramid (Kevlar) (sample 141)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
141-1	6.0320	5.9920	0.0400	0.66
141-2	5.1312	5.1002	0.0310	0.60
141-3	5.4897	5.4581	0.0316	0.58
average			0.0342	0.61
uncertainty			0.0125	0.11
SD			0.0050	0.04
RSD			14.71	7.26

40% melamine-60% p-aramid (Kevlar) (sample 142)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
142-1	5.7878	5.7443	0.0435	0.75
142-2	4.8355	4.8030	0.0325	0.67
142-3	5.3756	5.3384	0.0372	0.69
average			0.0377	0.71
uncertainty			0.0137	0.10
SD			0.0055	0.04
RSD			14.63	5.86

25% melamine-60% m-aramid (Conex) (sample 143)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
143-1	5.8209	5.7885	0.0324	0.56
143-2	5.6349	5.6047	0.0302	0.54
143-3	5.6817	5.6546	0.0271	0.48
average			0.0299	0.52
uncertainty			0.0066	0.10
SD			0.0027	0.04
RSD			8.91	7.90

17% melamine-17% modacrylic- 66% polyester (sample 144)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
144-1	5.9610	5.9443	0.0167	0.28
144-2	6.4777	6.4617	0.0160	0.25
144-3	5.9672	5.9493	0.0179	0.30
144-4	4.3673	4.3559	0.0114	0.26
144-5	4.5994	4.5862	0.0132	0.29
average			0.0150	0.28
uncertainty			0.0033	0.03
SD			0.0027	0.02
RSD			17.75	7.66

12% melamine -30% lyocell- 34 % modacrylic- 24% polyester (sample 145)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
145-1	4.2327	4.2131	0.0196	0.46
145-2	3.4765	3.4587	0.0178	0.51
145-3	4.2106	4.1931	0.0175	0.42
average			0.0183	0.46
uncertainty			0.0028	0.12
SD			0.0011	0.05
RSD			6.21	10.40

Pre-treatment with petroleum ether in Soxtec

100 % melamine (samples 136,137,171)

JRC code	sample mass	melamine mass	melamine mass loss	melamine mass loss
	g	g	g	%
136-1	0.9546	0.9498	0.0048	0.50
136-2	1.0262	1.0199	0.0063	0.61
136-3	1.1177	1.1090	0.0087	0.78
136-4	1.1896	1.1828	0.0068	0.57
136-5	1.0490	1.0430	0.0060	0.57
136-6	1.0390	1.0321	0.0069	0.66
136-7	0.9583	0.9507	0.0076	0.79
136-8	0.9698	0.9640	0.0058	0.60
136-9	0.9772	0.9691	0.0081	0.83
136-10	1.1649	1.1579	0.0070	0.60
136-11	2.0698	2.0511	0.0187	0.90
136-12	2.0403	2.0236	0.0167	0.82
136-13	2.0414	2.0284	0.0130	0.64
136-14	1.9903	1.9728	0.0175	0.88
136-15	1.9453	1.9302	0.0151	0.78
average			0.0099	0.70
uncertainty			0.0027	0.07
SD			0.0048	0.13
RSD			48.58	18.22

JRC code	sample mass	melamine mass	melamine mass loss	melamine mass loss
	g	g	g	%
137-1	1.2551	1.2471	0.0080	0.64
137-2	1.0391	1.0323	0.0068	0.65
137-3	0.9858	0.9792	0.0066	0.67
137-4	0.9547	0.9472	0.0075	0.79
137-5	0.9422	0.9354	0.0068	0.72
137-6	0.9913	0.9850	0.0063	0.64
137-7	0.9855	0.9795	0.0060	0.61
137-8	0.9908	0.9840	0.0068	0.69
137-9	1.1428	1.1317	0.0111	0.97
137-10	1.0760	1.0649	0.0111	1.03
average			0.0077	0.74
uncertainty			0.0013	0.11
SD			0.0019	0.15
RSD			24.39	19.87

JRC code	sample mass	melamine mass	melamine mass loss	melamine mass loss
	g	g	g	%
171-1	3.0457	3.0229	0.0228	0.75
171-2	2.4306	2.4134	0.0172	0.71
171-3	3.0574	3.0305	0.0269	0.88
average			0.0223	0.78
uncertainty			0.0121	0.22
SD			0.0049	0.09
RSD			21.84	11.55

melamine-viscose (sample 172)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
172-1	9.0138	8.9374	0.0764	0.85
172-2	9.5944	9.5193	0.0751	0.78
172-3	6.8314	6.7767	0.0547	0.80
average			0.0687	0.81
uncertainty			0.0302	0.08
SD			0.0122	0.03
RSD			17.71	4.13

melamine-Visil (sample 173)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
173-1	4.9117	4.8635	0.0482	0.98
173-2	6.5908	6.5268	0.0640	0.97
173-3	6.0568	5.9988	0.0580	0.96
average			0.0567	0.97
uncertainty			0.0198	0.03
SD			0.0080	0.01
RSD			14.06	1.23

melamine-polyester (sample 174)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
174-1	10.2346	10.1618	0.0728	0.71
174-2	10.0277	9.9628	0.0649	0.65
174-3	10.6326	10.5576	0.0750	0.71
average			0.0709	0.69
uncertainty			0.0132	0.09
SD			0.0053	0.04
RSD			7.49	5.15

melamine-cotton (sample 175)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
175-1	9.9316	9.8456	0.0860	0.87
175-2	11.4941	11.3948	0.0993	0.86
175-3	12.8045	12.6951	0.1094	0.85
average			0.0982	0.86
uncertainty			0.0292	0.02
SD			0.0117	0.01
RSD			11.95	0.72

melamine-viscose (sample 176)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
176-1	9.7096	9.6544	0.0552	0.57
176-2	8.4257	8.3716	0.0541	0.64
176-3	7.4267	7.3779	0.0488	0.66
average			**0.0527**	**0.62**
uncertainty			**0.0085**	**0.12**
SD			**0.0034**	**0.05**
RSD			**6.49**	**7.61**

melamine-m-aramid (Nomex) (sample 177)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
177-1	11.8415	11.7271	0.1144	0.97
177-2	10.2222	10.1283	0.0939	0.92
177-3	10.1522	10.0293	0.1229	1.21
177-4	6.7264	6.6220	0.1044	1.55
average			**0.1104**	**1.16**
uncertainty			**0.0237**	**0.46**
SD			**0.0149**	**0.29**
RSD			**13.50**	**24.95**

melamine-p-aramid (Kevlar) (sample 178)

JRC code	untreated sample mass	sample mass	sample mass loss	sample mass loss
	g	g	g	%
178-1	10.9792	10.8947	0.0845	0.77
178-2	10.0958	10.0093	0.0865	0.86
178-3	13.0314	12.8926	0.1388	1.07
178-4	7.7886	7.7146	0.0740	0.95
178-5	8.8572	8.7706	0.0866	0.98
average			**0.0941**	**0.92**
uncertainty			**0.0317**	**0.14**
SD			**0.0255**	**0.11**
RSD			**27.14**	**12.32**

Correction factors *d* according to the methods described on the Directive 96/73/EC

100% melamine (samples 136,137)

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M1-136-1	0.9739	0.9688	99.51	0.49	1.005
	M1-136-2	0.9670	0.9646	99.77	0.23	1.002
	M1-136-3	0.9776	0.9744	99.69	0.31	1.003
	M1-136-4	0.9610	0.9567	99.58	0.42	1.004
	M1-136-5	0.9689	0.9629	99.42	0.58	1.006
	M1-136-6	1.0205	1.0165	99.63	0.37	1.004
	M1-136-7	0.9822	0.9778	99.58	0.42	1.004
	M1-136-8	1.0027	0.9961	99.38	0.62	1.007
	M1-136-9	1.0109	1.0019	99.17	0.83	1.009
method 1	**M1-136-10**	1.0244	1.0182	99.43	0.57	1.006
	M1-137-1	1.0270	1.0205	99.41	0.59	1.006
	M1-137-2	0.9464	0.9401	99.38	0.62	1.007
	M1-137-3	1.0302	1.0244	99.47	0.53	1.006
	M1-137-4	1.0540	1.0479	99.46	0.54	1.006
	M1-137-5	1.0681	1.0602	99.31	0.69	1.007
	M1-137-6	0.9577	0.9526	99.50	0.50	1.005
	M1-137-7	1.0246	1.0179	99.39	0.61	1.007
	M1-137-8	1.1860	1.1771	99.30	0.70	1.008
	M1-137-9	1.0292	1.0237	99.50	0.50	1.005
	M1-137-10	1.0257	1.0185	99.34	0.66	1.007
	average			**99.46**	**0.54**	**1.006**
	uncertainty			**0.07**	**0.07**	**0.001**
	SD			**0.14**	**0.14**	**0.002**
	RSD%			**0.14**	**26.48**	**0.15**

method	JRC code	sample mass g	melamine mass g	melamine %	other %	d
	M2-136-1	1.2480	1.2367	99.15	0.85	1.009
	M2-136-2	1.1722	1.1632	99.28	0.72	1.008
	M2-136-3	1.1880	1.1772	99.15	0.85	1.009
	M2-136-4	1.0342	1.0280	99.44	0.56	1.006
	M2-136-5	1.4889	1.4754	99.15	0.85	1.009
	M2-136-6	1.1715	1.1646	99.45	0.55	1.006
	M2-136-7	0.9966	0.9918	99.55	0.45	1.005
	M2-136-8	1.0444	1.0382	99.44	0.56	1.006
	M2-136-9	1.0871	1.0766	99.10	0.90	1.010
method 2	M2-136-10	1.1430	1.1361	99.44	0.56	1.006
	M2-137-1	0.9907	0.9810	99.08	0.92	1.010
	M2-137-2	0.9480	0.9395	99.16	0.84	1.009
	M2-137-3	1.0093	1.0026	99.38	0.62	1.007
	M2-137-4	1.0091	1.0019	99.33	0.67	1.007
	M2-137-5	0.9737	0.9687	99.52	0.48	1.005
	M2-137-6	1.0029	0.9957	99.33	0.67	1.007
	M2-137-7	0.9530	0.9470	99.41	0.59	1.006
	M2-137-8	0.9494	0.9450	99.57	0.43	1.005
	M2-137-9	0.9613	0.9553	99.42	0.58	1.006
	M2-137-10	1.0103	1.0041	99.43	0.57	1.006
	average			99.34	0.66	1.007
	uncertainty			0.07	0.07	0.001
	SD			0.15	0.15	0.002
	RSD%			0.16	23.44	0.17

method	JRC code	sample mass g	melamine mass g	melamine %	other %	d
	M3-136-1	1.0490	1.0467	99.80	0.20	1.002
	M3-136-2	0.9341	0.9329	99.88	0.12	1.001
	M3-136-3	1.0066	1.0067	100.01	-0.01	1.000
	M3-136-4	1.0372	1.0349	99.79	0.21	1.002
	M3-136-5	0.8344	0.8352	100.09	-0.09	0.999
	M3-136-6	0.9846	0.9846	100.00	0.00	1.000
	M3-136-7	0.9393	0.9382	99.89	0.11	1.001
	M3-136-8	0.9851	0.9853	100.02	-0.02	1.000
	M3-136-9	1.2099	1.2108	100.07	-0.07	0.999
method 3	M3-136-10	0.6602	0.6595	99.90	0.10	1.001
	M3-137-1	1.0429	1.0420	99.92	0.08	1.001
	M3-137-2	1.0364	1.0358	99.95	0.05	1.001
	M3-137-3	1.0220	1.0223	100.03	-0.03	1.000
	M3-137-4	1.0492	1.0504	100.11	-0.11	0.999
	M3-137-5	0.9928	0.9935	100.07	-0.07	0.999
	M3-137-6	1.0586	1.0588	100.02	-0.02	1.000
	M3-137-7	0.9690	0.9685	99.95	0.05	1.001
	M3-137-8	1.0277	1.0319	100.38	-0.38	0.996
	M3-137-9	1.0634	1.0624	99.91	0.09	1.001
	M3-137-10	1.1120	1.1057	99.47	0.53	1.006
	average			99.96	0.04	1.000
	uncertainty			0.08	0.08	0.001
	SD			0.17	0.17	0.002
	RSD%			0.17	458.72	0.19

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M4-136-1	0.9285	0.9196	99.10	0.90	1.010
	M4-136-2	1.0038	0.9942	99.11	0.89	1.010
	M4-136-3	1.0934	1.0792	98.79	1.21	1.013
	M4-136-4	1.1646	1.1558	99.29	0.71	1.008
	M4-136-5	1.0105	1.0015	99.17	0.83	1.009
	M4-136-6	1.0151	1.0055	99.12	0.88	1.010
	M4-136-7	0.9419	0.9299	98.81	1.19	1.013
	M4-136-8	0.9526	0.9456	99.31	0.69	1.007
	M4-136-9	0.9518	0.9429	99.13	0.87	1.009
method 4	M4-136-10	1.1421	1.1350	99.42	0.58	1.006
	M4-137-1	1.1072	1.0968	99.12	0.88	1.009
	M4-137-2	1.0455	1.0387	99.39	0.61	1.007
	M4-137-3	1.0017	0.9952	99.39	0.61	1.007
	M4-137-4	1.0621	1.0550	99.37	0.63	1.007
	M4-137-5	1.0009	0.9890	98.89	1.11	1.012
	M4-137-6	1.0692	1.0608	99.27	0.73	1.008
	M4-137-7	1.0228	1.0139	99.19	0.81	1.009
	M4-137-8	1.1136	1.1033	99.14	0.86	1.009
	M4-137-9	1.0289	1.0222	99.39	0.61	1.007
	M4-137-10	0.8378	0.8300	99.13	0.87	1.009
	average			99.18	0.82	1.009
	uncertainty			0.09	0.09	0.001
	SD			0.19	0.19	0.002
	RSD%			0.19	22.84	0.20

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M5-136-1	0.9804	0.9762	99.60	0.40	1.004
	M5-136-2	0.9744	0.9703	99.61	0.39	1.004
	M5-136-3	1.0596	1.0558	99.66	0.34	1.004
	M5-136-4	0.9733	0.9703	99.71	0.29	1.003
	M5-136-5	0.9793	0.9772	99.80	0.20	1.002
	M5-136-6	0.9969	0.9914	99.48	0.52	1.006
	M5-136-7	1.0047	1.0008	99.64	0.36	1.004
	M5-136-8	0.9713	0.9660	99.49	0.51	1.005
	M5-136-9	1.0477	1.0448	99.74	0.26	1.003
method 5	M5-136-10	1.1147	1.1097	99.58	0.42	1.005
	M5-137-1	0.9654	0.9601	99.49	0.51	1.006
	M5-137-2	1.2056	1.1975	99.37	0.63	1.007
	M5-137-3	1.0216	1.0142	99.32	0.68	1.007
	M5-137-4	0.8965	0.8900	99.32	0.68	1.007
	M5-137-5	0.8573	0.8532	99.55	0.45	1.005
	M5-137-6	1.0825	1.0782	99.63	0.37	1.004
	M5-137-7	0.9835	0.9783	99.51	0.49	1.005
	M5-137-8	0.9314	0.9259	99.45	0.55	1.006
	M5-137-9	0.7653	0.7605	99.41	0.59	1.006
	M5-137-10	0.9072	0.9021	99.47	0.53	1.006
	average			99.54	0.46	1.005
	uncertainty			0.06	0.06	0.001
	SD			0.13	0.13	0.001
	RSD%			0.13	29.27	0.14

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M6-136-1	0.9946	0.9883	99.41	0.59	1.006
	M6-136-2	1.0140	1.0098	99.61	0.39	1.004
	M6-136-3	1.0613	1.0537	99.33	0.67	1.007
	M6-136-4	0.8920	0.8857	99.34	0.66	1.007
	M6-136-5	1.1041	1.0997	99.63	0.37	1.004
	M6-136-6	1.0484	1.0444	99.64	0.36	1.004
	M6-136-7	0.9064	0.9004	99.38	0.62	1.007
	M6-136-8	0.9860	0.9794	99.37	0.63	1.007
	M6-136-9	1.1416	1.1376	99.67	0.33	1.004
method 6	M6-136-10	1.0760	1.0727	99.71	0.29	1.003
	M6-137-1	0.9774	0.9743	99.70	0.30	1.003
	M6-137-2	0.9937	0.9874	99.41	0.59	1.006
	M6-137-3	0.9924	0.9880	99.59	0.41	1.004
	M6-137-4	0.9686	0.9654	99.69	0.31	1.003
	M6-137-5	0.9475	0.9442	99.67	0.33	1.003
	M6-137-6	0.9635	0.9571	99.38	0.62	1.007
	M6-137-7	0.9663	0.9619	99.57	0.43	1.005
	M6-137-8	0.9814	0.9779	99.67	0.33	1.004
	M6-137-9	0.9834	0.9811	99.78	0.22	1.002
	M6-137-10	0.9649	0.9600	99.53	0.47	1.005
	average			99.55	0.45	1.005
	uncertainty			0.07	0.07	0.001
	SD			0.15	0.15	0.002
	RSD%			0.15	32.99	0.16

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M7-136-1	1.1359	1.1276	99.30	0.70	1.007
	M7-136-2	1.0729	1.0432	99.12	0.88	1.009
	M7-136-3	0.9120	0.9053	99.30	0.70	1.007
	M7-136-4	1.0326	1.0240	99.21	0.79	1.008
	M7-136-5	1.0101	1.0028	99.31	0.69	1.007
	M7-137-1	1.2328	1.2223	99.19	0.81	1.009
	M7-137-2	1.1517	1.1430	99.28	0.72	1.008
	M7-137-3	0.9704	0.9601	98.99	1.01	1.011
	M7-137-4	1.0178	1.0038	98.69	1.31	1.014
method 7	M7-137-5	1.0092	0.9971	98.86	1.14	1.012
	M7-137-6	0.8672	0.8603	99.24	0.76	1.008
	M7-137-7	0.9454	0.9373	99.18	0.82	1.009
	M7-137-8	0.9204	0.9133	99.27	0.73	1.008
	M7-137-9	0.9532	0.9459	99.27	0.73	1.008
	M7-137-10	0.9738	0.9661	99.25	0.75	1.008
	M7-137-11	0.9936	0.9856	99.23	0.77	1.008
	M7-137-12	0.9367	0.9283	99.15	0.85	1.009
	M7-137-13	1.0020	0.9949	99.32	0.68	1.007
	M7-137-14	0.9196	0.9098	98.98	1.02	1.011
	M7-137-15	0.9641	0.9551	99.11	0.89	1.009
	average			99.16	0.84	1.009
	uncertainty			0.08	0.08	0.001
	SD			0.17	0.17	0.002
	RSD%			0.17	19.92	0.18

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M8-136-1	0.9821	0.9763	99.45	0.55	1.006
	M8-136-2	1.0527	1.0464	99.44	0.56	1.006
	M8-136-3	1.0319	1.0281	99.66	0.34	1.004
	M8-136-4	1.0378	1.0329	99.56	0.44	1.005
	M8-136-5	1.0163	1.0123	99.63	0.37	1.004
	M8-136-6	0.9584	0.9554	99.71	0.29	1.003
	M8-136-7	1.0507	1.0430	99.31	0.69	1.007
	M8-136-8	1.0774	1.0699	99.35	0.65	1.007
	M8-136-9	1.1642	1.1562	99.36	0.64	1.007
method 8	M8-136-10	0.9965	0.9905	99.44	0.56	1.006
	M8-137-1	0.9687	0.9629	99.44	0.56	1.006
	M8-137-2	0.9797	0.9732	99.38	0.62	1.007
	M8-137-3	0.9884	0.9833	99.52	0.48	1.005
	M8-137-4	1.0610	1.0547	99.44	0.56	1.006
	M8-137-5	1.1798	1.1708	99.29	0.71	1.008
	M8-137-6	1.0390	1.0311	99.29	0.71	1.008
	M8-137-7	1.0670	1.0580	99.21	0.79	1.009
	M8-137-8	0.9415	0.9352	99.37	0.63	1.007
	M8-137-9	1.0217	1.0140	99.30	0.70	1.008
	M8-137-10	1.0300	1.0240	99.46	0.54	1.006
	average			99.43	0.57	1.006
	uncertainty			0.06	0.06	0.001
	SD			0.13	0.13	0.001
	RSD%			0.13	23.16	0.14

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M9-136-1	1.1106	1.1045	99.49	0.51	1.006
	M9-136-2	1.0206	1.0140	99.40	0.60	1.007
	M9-136-3	0.9046	0.8973	99.25	0.75	1.008
	M9-136-4	0.5093	0.5063	99.45	0.55	1.006
	M9-136-5	0.5867	0.5838	99.54	0.46	1.005
	M9-136-6	0.5478	0.5440	99.35	0.65	1.007
	M9-136-7	0.5156	0.5125	99.44	0.56	1.006
	M9-136-8	0.9834	0.9765	99.34	0.66	1.007
	M9-136-9	1.2099	1.2000	99.23	0.77	1.008
method 9	M9-136-10	0.6602	0.6551	99.28	0.72	1.008
	M9-137-1	0.9994	0.9893	99.05	0.95	1.010
	M9-137-2	1.0089	1.0005	99.22	0.78	1.008
	M9-137-3	1.0437	1.0348	99.20	0.80	1.009
	M9-137-4	1.0287	1.0181	99.04	0.96	1.010
	M9-137-5	0.9773	0.9665	98.97	1.03	1.011
	M9-137-6	1.0056	0.9939	98.91	1.09	1.012
	M9-137-7	1.1074	1.0950	98.95	1.05	1.011
	M9-137-8	1.1355	1.1226	98.94	1.06	1.011
	M9-137-9	1.0040	0.9913	98.82	1.18	1.013
	M9-137-10	1.0050	0.9943	99.00	1.00	1.011
	average			99.19	0.81	1.009
	uncertainty			0.10	0.10	0.001
	SD			0.22	0.22	0.002
	RSD%			0.22	27.07	0.24

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M10-136-1	1.0013	0.9968	99.58	0.42	1.005
	M10-136-2	1.0088	1.0033	99.49	0.51	1.005
	M10-136-3	0.9918	0.9918	100.00	0.00	1.000
	M10-136-4	1.0965	1.0959	99.95	0.05	1.001
	M10-136-5	1.0237	1.0232	99.95	0.05	1.000
	M10-136-6	1.0220	1.0195	99.77	0.23	1.002
	M10-136-7	1.0557	1.0555	99.98	0.02	1.000
	M10-136-8	0.9753	0.9750	99.97	0.03	1.000
	M10-136-9	1.0645	1.0642	99.97	0.03	1.000
method 10	M10-136-10	0.9639	0.9631	99.92	0.08	1.001
	M10-137-1	1.1056	1.1025	99.74	0.26	1.003
	M10-137-2	0.9828	0.9824	99.96	0.04	1.000
	M10-137-3	0.9189	0.9177	99.88	0.12	1.001
	M10-137-4	0.8848	0.8834	99.85	0.15	1.002
	M10-137-5	0.9737	0.9714	99.78	0.22	1.002
	M10-137-6	0.9512	0.9501	99.89	0.11	1.001
	M10-137-7	0.8701	0.8690	99.88	0.12	1.001
	M10-137-8	1.0600	1.0605	100.04	-0.04	1.000
	M10-137-9	0.9432	0.9421	99.89	0.11	1.001
	M10-137-10	0.9929	0.9915	99.87	0.13	1.001
	average			99.87	0.13	1.001
	uncertainty			0.07	0.07	0.001
	SD			0.14	0.14	0.002
	RSD%			0.14	106.63	0.15

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M11-136-1	1.0472	1.0470	99.98	0.02	1.000
	M11-136-2	1.1438	1.1436	99.98	0.02	1.000
	M11-136-3	1.0518	1.0508	99.91	0.09	1.001
	M11-136-4	0.9672	0.9676	100.04	-0.04	1.000
	M11-136-5	0.9356	0.9360	100.04	-0.04	1.000
	M11-136-6	1.0349	1.0333	99.86	0.14	1.002
	M11-136-7	1.1102	1.1105	100.03	-0.03	1.000
	M11-136-8	1.0407	1.0406	99.99	0.01	1.000
	M11-136-9	0.9608	0.9595	99.87	0.13	1.001
method 11	M11-136-10	1.0579	1.0558	99.81	0.19	1.002
	M11-137-1	1.1986	1.1886	99.22	0.78	1.008
	M11-137-2	1.0090	0.9977	98.95	1.05	1.011
	M11-137-3	0.9553	0.9460	99.09	0.91	1.010
	M11-137-4	0.9331	0.9243	99.12	0.88	1.010
	M11-137-5	0.9224	0.9126	99.01	0.99	1.011
	M11-137-6	0.9676	0.9581	99.08	0.92	1.010
	M11-137-7	0.9610	0.9526	99.18	0.82	1.009
	M11-137-8	0.9680	0.9583	99.06	0.94	1.010
	M11-137-9	1.1167	1.1068	99.17	0.83	1.009
	M11-137-10	1.0511	1.0412	99.12	0.88	1.010
	average			99.53	0.47	1.005
	uncertainty			0.21	0.21	0.002
	SD			0.44	0.44	0.005
	RSD%			0.45	93.61	0.48

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M13-136-1	1.0410	1.0318	99.17	0.83	1.009
	M13-136-2	0.9938	0.9865	99.31	0.69	1.007
	M13-136-3	1.0232	1.0137	99.13	0.87	1.009
	M13-136-4	0.9922	0.9839	99.22	0.78	1.008
	M13-136-5	0.9801	0.9705	99.08	0.92	1.010
	M13-136-6	1.0062	1.0016	99.57	0.43	1.005
	M13-136-7	1.0036	0.9990	99.57	0.43	1.005
	M13-136-8	1.1498	1.1403	99.23	0.77	1.008
	M13-136-9	0.9814	0.9720	99.10	0.90	1.010
method 13	M13-136-10	0.9650	0.9576	99.28	0.72	1.008
	M13-137-1	0.9887	0.9787	99.05	0.95	1.010
	M13-137-2	0.9797	0.9702	99.09	0.91	1.010
	M13-137-3	0.9884	0.9824	99.43	0.57	1.006
	M13-137-4	1.0510	1.0435	99.33	0.67	1.007
	M13-137-5	1.1566	1.1495	99.43	0.57	1.006
	M13-137-6	1.0070	1.0007	99.42	0.58	1.006
	M13-137-7	1.0147	1.0069	99.28	0.72	1.008
	M13-137-8	0.9715	0.9654	99.41	0.59	1.006
	M13-137-9	1.1617	1.1526	99.27	0.73	1.008
	M13-137-10	0.9990	0.9915	99.30	0.70	1.008
	average			99.28	0.72	1.008
	uncertainty			0.07	0.07	0.001
	SD			0.15	0.15	0.002
	RSD%			0.16	21.52	0.17

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M14-136-1	0.9818	0.9765	99.50	0.50	1.005
	M14-136-2	0.9455	0.9408	99.54	0.46	1.005
	M14-136-3	1.1290	1.1185	99.13	0.87	1.009
	M14-136-4	0.9927	0.9836	99.14	0.86	1.009
	M14-136-5	1.1992	1.1930	99.52	0.48	1.005
	M14-136-6	1.0218	1.0138	99.27	0.73	1.008
	M14-136-7	0.9845	0.9795	99.53	0.47	1.005
	M14-136-8	0.9461	0.9375	99.15	0.85	1.009
	M14-136-9	0.9815	0.9723	99.12	0.88	1.009
method 14	M14-136-10	0.9923	0.9835	99.17	0.83	1.009
	M14-137-1	1.0033	0.9941	99.14	0.86	1.009
	M14-137-2	0.9681	0.9595	99.17	0.83	1.009
	M14-137-3	0.9658	0.9571	99.16	0.84	1.009
	M14-137-4	0.9960	0.9897	99.41	0.59	1.006
	M14-137-5	0.9953	0.9901	99.51	0.49	1.005
	M14-137-6	0.9548	0.9505	99.58	0.42	1.005
	M14-137-7	1.0027	0.9956	99.34	0.66	1.007
	M14-137-8	1.0110	1.0047	99.42	0.58	1.006
	M14-137-9	1.0329	1.0239	99.19	0.81	1.009
	M14-137-10	0.9823	0.9754	99.34	0.66	1.007
	average			99.32	0.68	1.007
	uncertainty			0.08	0.08	0.001
	SD			0.17	0.17	0.002
	RSD%			0.17	24.59	0.18

method	JRC code	sample mass	melamine mass	melamine	other	d
		g	g	%	%	
	M15-136-1	0.8871	0.8795	99.20	0.80	1.009
	M15-136-2	0.8849	0.8768	99.14	0.86	1.009
	M15-136-3	0.8849	0.8749	98.94	1.06	1.011
	M15-136-4	0.8314	0.8242	99.19	0.81	1.009
	M15-136-5	0.8422	0.8358	99.29	0.71	1.008
	M15-136-6	1.0538	1.0410	98.86	1.14	1.012
	M15-136-7	0.9561	0.9457	98.98	1.02	1.011
	M15-136-8	0.9750	0.9657	99.11	0.89	1.010
	M15-136-9	0.9607	0.9516	99.11	0.89	1.010
method 15	**M15-136-10**	1.0486	1.0363	98.90	1.10	1.012
	M15-137-1	0.9472	0.9354	98.83	1.17	1.013
	M15-137-2	0.8907	0.8797	98.84	1.16	1.013
	M15-137-3	0.9133	0.9010	98.74	1.26	1.014
	M15-137-4	0.9676	0.9571	98.99	1.01	1.011
	M15-137-5	0.8736	0.8618	98.74	1.26	1.014
	M15-137-6	0.9518	0.9450	99.33	0.67	1.007
	M15-137-7	0.8825	0.8729	98.98	1.02	1.011
	M15-137-8	0.9659	0.9576	99.20	0.80	1.009
	M15-137-9	0.9775	0.9715	99.43	0.57	1.006
	M15-137-10	0.8948	0.8898	99.48	0.52	1.006
	average			**99.06**	**0.94**	**1.010**
	uncertainty			**0.10**	**0.10**	**0.001**
	SD			**0.22**	**0.22**	**0.002**
	RSD%			**0.22**	**23.27**	**0.23**

100% lyocell (Tencel) (sample 152)

method	JRC code	sample mass	residue mass	lyocell	other	d
		g	g	%	%	
	M8-152-1	0.9171	0.9155	99.85	0.15	1.002
	M8-152-2	0.9978	0.9967	99.90	0.10	1.001
	M8-152-3	1.0322	1.0316	99.95	0.05	1.001
	M8-152-4	0.7891	0.7884	99.92	0.08	1.001
	M8-152-5	0.7430	0.7418	99.86	0.14	1.002
	M8-152-6	0.8829	0.8808	99.79	0.21	1.002
	M8-152-7	0.9164	0.9159	99.95	0.05	1.001
	M8-152-8	0.9921	0.9908	99.88	0.12	1.001
	M8-152-9	0.9024	0.9000	99.76	0.24	1.003
method 8	**M8-152-10**	1.0009	0.9978	99.73	0.27	1.003
	M8-152-11	0.9316	0.9298	99.83	0.17	1.002
	M8-152-12	0.8894	0.8880	99.86	0.14	1.002
	M8-152-13	0.9313	0.9303	99.90	0.10	1.001
	M8-152-14	1.0658	1.0648	99.92	0.08	1.001
	M8-152-15	0.9915	0.9896	99.83	0.17	1.002
	M8-152-16	1.0658	1.0646	99.90	0.10	1.001
	M8-152-17	1.0866	1.0838	99.77	0.23	1.003
	M8-152-18	1.1260	1.1249	99.91	0.09	1.001
	M8-152-19	0.9990	0.9957	99.71	0.29	1.003
	M8-152-20	1.0078	1.0045	99.71	0.29	1.003
	average			**99.85**	**0.15**	**1.002**
	uncertainty			**0.04**	**0.04**	**0.000**
	SD			**0.08**	**0.08**	**0.001**
	RSD%			**0.08**	**51.07**	**0.089**

100% modacrylic (sample 154)

method	JRC code	sample mass	residue mass	modacrylic	other	d
		g	g	%	%	
	M7-154-1	1.0365	1.0271	99.11	0.89	1.009
	M7-154-2	1.0423	1.0344	99.26	0.74	1.008
	M7-154-3	1.0269	1.0203	99.37	0.63	1.006
	M7-154-4	1.0552	1.0503	99.54	0.46	1.005
	M7-154-5	1.0037	0.9975	99.39	0.61	1.006
	M7-154-6	0.9770	0.9702	99.32	0.68	1.007
	M7-154-7	1.0338	1.0330	99.92	0.08	1.001
	M7-154-8	0.9701	0.9644	99.42	0.58	1.006
	M7-154-9	0.9021	0.8962	99.36	0.64	1.007
method 7	M7-154-10	1.0532	1.0475	99.47	0.53	1.005
	M7-154-11	0.9744	0.9684	99.40	0.60	1.006
	M7-154-12	0.9067	0.9004	99.32	0.68	1.007
	M7-154-13	1.0147	1.0065	99.21	0.79	1.008
	M7-154-14	0.9303	0.9247	99.41	0.59	1.006
	M7-154-15	1.0036	0.9957	99.23	0.77	1.008
	M7-154-16	0.9813	0.9758	99.45	0.55	1.006
	M7-154-17	1.0060	0.9976	99.18	0.82	1.008
	M7-154-18	1.0474	1.0405	99.35	0.65	1.007
	M7-154-19	1.0319	1.0222	99.08	0.92	1.009
	M7-154-20	1.0588	1.0500	99.19	0.81	1.008
	average			99.35	0.65	1.007
	uncertainty			0.09	0.09	0.001
	SD			0.18	0.18	0.002
	RSD%			0.18	28.15	0.188

100% m-aramid (Nomex) (sample 056)

method	JRC code	sample mass	residue mass	m-aramid	other
		g	g	%	%
	M14-056-1	0.8514	0.0175	98.09	1.91
	M14-056-2	0.7762	0.0112	98.66	1.34
	M14-056-3	0.9819	0.0122	98.85	1.15
	M14-056-4	1.0199	0.0118	98.93	1.07
method 14	M14-056-5	0.9156	0.0201	97.96	2.04
	M14-056-6	0.7778	0.0157	98.13	1.87
	M14-056-7	0.7563	0.0176	97.84	2.16
	M14-056-8	0.8200	0.0156	98.24	1.76
	M14-056-9	0.9527	0.0181	98.24	1.76
	M14-056-10	0.8926	0.0122	98.73	1.27
	average			98.37	1.63
	uncertainty			0.28	0.28
	SD			0.39	0.39
	RSD%			0.40	23.91

100% p-aramid (Kevlar) (sample 057)

method	JRC code	sample mass	residue mass	p-aramid	other
		g	g	%	%
	M14-057-1	0.8767	0.0175	98.15	1.85
	M14-057-2	0.9322	0.017	98.31	1.69
	M14-057-3	0.8957	0.0204	97.89	2.11
	M14-057-4	0.9399	0.0145	98.57	1.43
method 14	M14-057-5	0.7114	0.0167	97.82	2.18
	M14-057-6	0.7812	0.0158	98.12	1.88
	M14-057-7	0.7595	0.0129	98.43	1.57
	M14-057-8	0.9442	0.0216	97.88	2.12
	M14-057-9	0.8843	0.0191	98.00	2.00
	M14-057-10	0.8882	0.0184	98.08	1.92
	average			98.12	1.88
	uncertainty			0.18	0.18
	SD			0.25	0.25
	RSD%			0.25	13.18

Correction factors *d* according to the methods proposed by the applicant

Method using 50 % m/m H_2SO_4, 90°C-(contact time: 3 hours)

100% melamine (sample 136)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
136-1	0.8938	0.0004	99.96	0.04
136-2	0.9080	0.0002	99.98	0.02
136-3	1.0220	0.0002	99.98	0.02
136-4	0.8840	0.0003	99.97	0.03
136-5	1.0170	0.0003	99.97	0.03
136-6	0.8968	0.0004	99.96	0.04
136-7	0.9429	0.0003	99.97	0.03
136-8	0.8705	0.0005	99.95	0.05
136-9	0.9504	0.0002	99.98	0.02
136-10	1.0419	0.0003	99.97	0.03
average			**99.97**	**0.03**
uncertainty			**0.01**	**0.01**
SD			**0.01**	**0.01**
RSD%			**0.01**	**36.66**

Method using 50 % m/m H2SO4, 90°C-(contact time: 2.5 hours)

100% melamine (sample 137)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
137-1	0.9385	0.0004	99.96	0.04
137-2	0.9795	0.0003	99.97	0.03
137-3	1.0077	0.0009	99.92	0.08
137-4	1.0010	0.0009	99.92	0.08
137-5	0.8264	0.0003	99.97	0.03
average			**99.95**	**0.05**
uncertainty			**0.03**	**0.03**
SD			**0.03**	**0.03**
RSD%			**0.03**	**50.89**

Method using 50 % m/m H2SO4, 90°C-(contact time: 30 minutes)

100% melamine (sample 136)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
137-6	0.9253	0.0001	99.99	0.01
137-7	1.0075	0.0007	99.94	0.06
137-8	0.8709	0.0001	99.99	0.01
137-9	0.9363	0.0004	99.96	0.04
137-10	1.0685	0.0002	99.98	0.02
average			**99.97**	**0.03**
uncertainty			**0.03**	**0.03**
SD			**0.02**	**0.02**
RSD%			**0.02**	**82.51**

Method using 50 % m/m H2SO4, 90°C-(contact time: 1 hour)

100% melamine (sample 136)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
136-1	1.0682	0.0052	99.54	0.46
136-2	0.9626	0.0042	99.59	0.41
136-3	0.9584	0.0058	99.43	0.57
136-4	0.9894	0.0037	99.65	0.35
136-5	0.9615	0.0015	99.85	0.15
136-6	0.9885	0.0086	99.19	0.81
136-7	1.0452	0.0066	99.41	0.59
136-8	0.9722	0.0042	99.60	0.40
136-9	0.9593	0.0030	99.71	0.29
136-10	0.9777	0.0056	99.46	0.54
average			**99.54**	**0.46**
uncertainty			**0.13**	**0.13**
SD			**0.18**	**0.18**
RSD%			**0.18**	**40.28**

100% melamine (sample 171)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
171-1	0.8581	-0.0017	100.19	-0.19
171-2	0.8567	0.0000	100.00	0.00
171-3	0.8677	0.0014	99.85	0.15
171-4	0.8073	0.0011	99.87	0.13
171-5	0.9701	0.0017	99.84	0.16
average			99.95	0.05
uncertainty			0.18	0.18
SD			0.15	0.15
RSD%			0.15	286.90

100% polyester (sample 153)

JRC code	sample mass	residue mass	PES	other	d
	g	g	%	%	
153-1	0.9731	0.9724	99.93	0.07	1.001
153-2	1.1778	1.1791	100.11	-0.11	0.999
153-3	1.0876	1.0852	99.78	0.22	1.002
153-4	1.1907	1.1910	100.02	-0.02	1.000
153-5	1.0459	1.0447	99.89	0.11	1.001
153-6	0.8798	0.8783	99.83	0.17	1.002
153-7	1.0372	1.0368	99.96	0.04	1.000
153-8	1.0522	1.0515	99.93	0.07	1.001
153-9	1.1003	1.1002	99.99	0.01	1.000
153-10	0.8673	0.8653	99.77	0.23	1.002
average			99.92	0.08	1.001
uncertainty			0.08	0.08	0.001
SD			0.11	0.11	0.001
RSD%			0.11	137.98	0.11

100% modacrylic (sample 154)

JRC code	sample mass	residue mass	modacrylic	other	d
	g	g	%	%	
154-1	1.0290	1.0271	99.82	0.18	1.002
154-2	1.0339	1.0322	99.84	0.16	1.002
154-3	0.8349	0.8341	99.91	0.09	1.001
154-4	1.0875	1.0845	99.73	0.27	1.003
154-5	0.8506	0.8484	99.75	0.25	1.003
154-6	1.0669	1.0638	99.72	0.28	1.003
154-7	1.0745	1.0705	99.64	0.36	1.004
154-8	1.1335	1.1311	99.79	0.21	1.002
154-9	0.9959	0.9920	99.62	0.38	1.004
154-10	1.2089	1.2064	99.80	0.20	1.002
average			99.76	0.24	1.002
uncertainty			0.06	0.06	0.001
SD			0.09	0.09	0.001
RSD%			0.09	37.44	0.09

100% lyocell (sample 152)

JRC code	sample mass	residue mass	lyocell	other
	g	g	%	%
152-1	0.8380	0.0079	99.16	0.84
152-2	0.8391	0.0068	99.28	0.72
152-3	0.8473	0.0077	99.19	0.81
152-4	0.8799	0.0074	99.26	0.74
152-5	0.9443	0.0082	99.23	0.77
152-6	0.8412	0.0074	99.22	0.78
152-7	0.7636	0.0069	99.20	0.80
152-8	0.9339	0.0071	99.32	0.68
152-9	1.0700	0.0089	99.26	0.74
152-10	0.9794	0.0075	99.32	0.68
average			**99.25**	**0.75**
uncertainty			**0.04**	**0.04**
SD			**0.05**	**0.05**
RSD%			**0.05**	**7.08**

100% cotton (sample 159/005)

JRC code	sample mass	residue mass	cotton	other
	g	g	%	%
159-1	0.9313	0.0071	99.30	0.70
159-2	0.9957	0.0080	99.26	0.74
159-3	1.0067	0.0074	99.32	0.68
005-4	0.7494	0.0056	99.31	0.69
005-5	0.9460	0.0069	99.33	0.67
average			**99.30**	**0.70**
uncertainty			**0.03**	**0.03**
SD			**0.03**	**0.03**
RSD%			**0.03**	**3.93**

100% viscose (sample 034)

JRC code	sample mass	residue mass	viscose	other
	g	g	%	%
034-1	1.0268	0.0062	99.47	0.53
034-2	0.9714	0.0056	99.49	0.51
034-3	1.0103	0.0061	99.47	0.53
034-4	1.0286	0.0056	99.52	0.48
034-5	1.0708	0.0059	99.51	0.49
average			**99.49**	**0.51**
uncertainty			**0.03**	**0.03**
SD			**0.02**	**0.02**
RSD%			**0.03**	**4.89**

100% polyamide (sample 038)

JRC code	sample mass	residue mass	polyamide	other
	g	g	%	%
038-1	1.0127	0.0015	99.86	0.14
038-2	1.0455	0.0033	99.70	0.30
038-3	1.0572	0.0022	99.80	0.20
038-4	1.1143	0.0017	99.86	0.14
038-5	1.1546	0.0046	99.62	0.38
average			**99.77**	**0.23**
uncertainty			**0.13**	**0.13**
SD			**0.10**	**0.10**
RSD%			**0.10**	**44.71**

100% triacetate (sample 040)

JRC code	sample mass	residue mass	triacetate	other
	g	g	%	%
040-1	1.0884	0.0022	99.81	0.19
040-2	1.0885	0.0025	99.79	0.21
040-3	0.9719	0.0017	99.84	0.16
040-4	0.9931	0.0020	99.81	0.19
040-5	0.9787	0.0020	99.81	0.19
average			**99.81**	**0.19**
uncertainty			**0.02**	**0.02**
SD			**0.02**	**0.02**
RSD%			**0.02**	**9.58**

100% p-aramid (Kevlar) (sample 150)

JRC code	sample mass	residue mass	Kevlar	other	d
	g	g	%	%	
150-1	1.1206	1.1127	99.35	0.65	1.007
150-2	1.0316	1.0236	99.28	0.72	1.008
150-3	1.2389	1.2304	99.36	0.64	1.007
150-4	1.0426	1.0349	99.32	0.68	1.007
150-5	1.0824	1.0734	99.23	0.77	1.008
150-6	1.0883	1.0805	99.34	0.66	1.007
150-7	1.0345	1.0265	99.28	0.72	1.008
150-8	1.0362	1.0288	99.34	0.66	1.007
150-9	1.0208	1.0143	99.41	0.59	1.006
150-10	1.0904	1.0823	99.31	0.69	1.007
average			**99.32**	**0.68**	**1.007**
uncertainty			**0.00**	**0.00**	**0.000**
SD			**0.05**	**0.05**	**0.001**
RSD%			**0.05**	**7.36**	**0.05**

100% m-aramid (Nomex) (sample 151)

JRC code	sample mass	residue mass	Nomex	other	d
	g	g	%	%	
151-1	1.1847	1.1757	99.30	0.70	1.008
151-2	1.2469	1.2388	99.40	0.60	1.007
151-3	1.1063	1.0985	99.35	0.65	1.007
151-4	1.0035	0.9952	99.23	0.77	1.008
151-5	0.9778	0.9704	99.30	0.70	1.008
151-6	0.9586	0.9514	99.30	0.70	1.008
151-7	0.9672	0.9610	99.41	0.59	1.006
151-8	1.0364	1.0290	99.34	0.66	1.007
151-9	0.9704	0.9640	99.39	0.61	1.007
151-10	1.0483	1.0423	99.47	0.53	1.006
average			**99.35**	**0.65**	**1.007**
uncertainty			**0.00**	**0.00**	**0.001**
SD			**0.07**	**0.07**	**0.001**
RSD%			**0.07**	**10.56**	**0.07**

Method using 90% m/m HCOOH, 90°C-(contact time: 3 hours)

100% melamine (sample 136)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
136-1	0.8924	0.0003	99.97	0.03
136-2	0.9066	0.0004	99.96	0.04
136-3	0.9649	0.0005	99.95	0.05
136-4	0.9756	0.0004	99.96	0.04
136-5	0.9612	0.0001	99.99	0.01
136-6	0.8523	0.0006	99.93	0.07
136-7	0.9050	0.0002	99.98	0.02
136-8	0.8667	0.0003	99.97	0.03
136-9	0.7250	0.0006	99.92	0.08
136-10	1.0379	0.0001	99.99	0.01
average			**99.96**	**0.04**
uncertainty			**0.02**	**0.02**
SD			**0.02**	**0.02**
RSD%			**0.02**	**59.43**

Method using 90% m/m HCOOH, 90°C-(contact time: 1 hour)

100% melamine (samples 136,137)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
137-1	0.7939	0.0005	99.94	0.06
137-2	0.8839	0.0003	99.97	0.03
137-3	0.9145	0.0006	99.94	0.06
137-4	0.9196	0.0004	99.96	0.04
137-5	0.9641	0.0005	99.95	0.05
136-1	0.9708	0.0005	99.95	0.05
136-2	0.9665	0.0008	99.92	0.08
136-3	0.9847	0.0006	99.94	0.06
136-4	0.9861	0.0004	99.96	0.04
136-5	0.9640	0.0010	99.90	0.10
average			**99.94**	**0.06**
uncertainty			**0.01**	**0.01**
SD			**0.02**	**0.02**
RSD%			**0.02**	**35.02**

100% melamine (sample 171)

JRC code	sample mass	residue mass	melamine	other
	g	g	%	%
171-1	0.8353	0.0005	99.94	0.06
171-2	0.9739	0.0001	99.99	0.01
171-3	0.7423	0.0015	99.81	0.19
171-4	0.6332	0.0001	99.99	0.01
171-5	0.5150	0.0008	99.85	0.15
average			99.92	0.08
uncertainty			0.10	0.10
SD			0.08	0.08
RSD%			0.08	97.01

100% p-aramid (Kevlar) (sample 150)

JRC code	sample mass	residue mass	Kevlar	other	d
	g	g	%	%	
056-1	0.8578	0.8459	98.71	1.29	1.014
056-2	0.8301	0.8175	98.59	1.41	1.015
056-3	0.9752	0.9604	98.59	1.41	1.015
056-4	0.8796	0.8668	98.65	1.35	1.015
056-5	0.9156	0.9037	98.80	1.20	1.013
056-6	0.9524	0.9388	98.68	1.32	1.014
056-7	0.8966	0.8840	98.70	1.30	1.014
056-8	1.0135	0.9995	98.72	1.28	1.014
150-1	0.8562	0.8452	98.81	1.19	1.013
150-2	0.9940	0.9829	98.97	1.03	1.011
average			98.72	1.28	1.014
uncertainty			0.08	0.08	0.001
SD			0.11	0.11	0.001
RSD%			0.11	8.77	0.12

100% m-aramid (Nomex) (sample 151)

JRC code	sample mass	residue mass	Nomex	other	d
	g	g	%	%	
151-1	0.9640	0.9529	98.93	1.07	1.012
151-2	1.0054	0.9931	98.87	1.13	1.012
151-3	0.9760	0.9633	98.79	1.21	1.013
151-4	1.0569	1.0407	98.58	1.42	1.016
151-5	1.0041	0.9907	98.76	1.24	1.014
151-6	1.0078	0.9942	98.75	1.25	1.014
151-7	0.9632	0.9506	98.79	1.21	1.013
151-8	0.9871	0.9734	98.71	1.29	1.014
average			98.77	1.23	1.013
uncertainty			0.19	0.19	0.002
SD			0.10	0.10	0.00
RSD%			0.11	8.53	0.11

100% lyocell (sample 152)

JRC code	sample mass	residue mass	lyocell	other	d
	g	g	%	%	
152-1	1.1455	1.0857	95.35	4.65	1.055
152-2	1.1322	1.0734	95.38	4.62	1.055
152-3	0.9171	0.8649	94.93	5.07	1.060
152-4	0.9898	0.9332	94.91	5.09	1.061
152-5	1.1047	1.0428	95.01	4.99	1.059
average			**95.11**	**4.89**	**1.058**
uncertainty			**0.29**	**0.29**	**0.004**
SD			**0.23**	**0.23**	**0.003**
RSD%			**0.24**	**4.73**	**0.27**

100% polyester (sample 153)

JRC code	sample mass	residue mass	PES	other	d
	g	g	%	%	
153-1	0.9392	0.8492	90.55	9.45	1.106
153-2	0.9890	0.8951	90.63	9.37	1.105
153-3	0.9490	0.8605	90.80	9.20	1.103
153-4	1.0339	0.9343	90.50	9.50	1.107
153-5	0.9845	0.8923	90.76	9.24	1.103
average			**90.65**	**9.35**	**1.105**
uncertainty			**0.16**	**0.16**	**0.002**
SD			**0.13**	**0.13**	**0.002**
RSD%			**0.15**	**1.41**	**0.15**

100% modacrylic (sample 154)

JRC code	sample mass	residue mass	modacrylic	other	d
	g	g	%	%	
154-1	0.9718	0.7947	82.07	17.93	1.223
154-2	0.9549	0.7670	80.63	19.37	1.245
154-3	1.0148	0.8174	80.86	19.14	1.241
154-4	1.0155	0.8247	81.51	18.49	1.231
154-5	0.9409	0.7746	82.61	17.39	1.215
154-6	0.9426	0.7617	81.11	18.89	1.237
154-7	1.0055	0.8281	82.64	17.36	1.214
average			**81.63**	**18.37**	**1.230**
uncertainty			**0.76**	**0.76**	**0.012**
SD			**0.82**	**0.82**	**0.013**
RSD%			**1.01**	**4.48**	**1.02**

100% polyamide (sample 038)

JRC code	sample mass	residue mass	polyamide	other
	g	g	%	%
038-1	0.9684	0.0024	99.77	0.23
038-2	0.9405	0.0000	100.00	0.00
038-3	1.0411	0.0012	99.89	0.11
038-4	1.0320	0.0007	99.94	0.06
038-5	0.9857	0.0029	99.72	0.28
average			**99.86**	**0.14**
uncertainty			**0.14**	**0.14**
SD			**0.12**	**0.12**
RSD%			**0.12**	**84.95**

100% cotton (sample 159)

JRC code	sample mass	residue mass	cotton	other	d
	g	g	%	%	
159-1	1.1274	1.1226	99.61	0.39	1.004
159-2	1.0677	1.0649	99.76	0.24	1.003
159-3	0.9996	0.9955	99.62	0.38	1.004
159-4	0.9334	0.9299	99.65	0.35	1.004
159-5	1.0435	1.0392	99.62	0.38	1.004
average			**99.65**	**0.35**	**1.004**
uncertainty			**0.08**	**0.08**	**0.001**
SD			**0.06**	**0.06**	**0.00**
RSD%			**0.06**	**17.74**	**0.07**

100% viscose (sample 034)

JRC code	sample mass	residue mass	viscose	other	d
	g	g	%	%	
034-1	0.8534	0.5220	64.03	35.97	1.635
034-2	0.9037	0.5517	63.91	36.09	1.638
034-3	0.8645	0.5273	63.86	36.14	1.639
034-4	0.9213	0.5640	64.08	35.92	1.634
034-5	0.9392	0.5739	63.97	36.03	1.637
average			**63.97**	**36.03**	**1.636**
uncertainty			**0.11**	**0.11**	**0.003**
SD			**0.09**	**0.09**	**0.00**
RSD%			**0.14**	**0.24**	**0.15**

Method using 6.15% m/m NaClO, 90°C-(contact time: 3 hours)

100% melamine (samples 136,137)

JRC code	sample mass	residue mass	melamine	other	d
	g	g	%	%	
137-1	1.1020	1.0284	93.73	6.27	1.072
137-2	1.1234	1.0469	93.61	6.39	1.073
137-3	1.0429	0.9720	93.62	6.38	1.073
137-4	1.0850	1.0166	94.08	5.92	1.067
137-5	1.0724	1.0044	94.05	5.95	1.068
137-6	1.1704	1.0926	93.76	6.24	1.071
136-1	1.0926	1.0148	93.31	6.69	1.077
136-2	0.9626	0.8934	93.25	6.75	1.077
136-3	0.9998	0.9267	93.13	6.87	1.079
136-4	0.6851	0.6339	92.98	7.02	1.081
average			**93.55**	**6.45**	**1.074**
uncertainty			**0.26**	**0.26**	**0.003**
SD			**0.37**	**0.37**	**0.005**
RSD%			**0.40**	**5.81**	**0.43**

Method using 6.15% m/m NaClO, 90°C-(contact time: 1 hour)

100% melamine (sample 136)

JRC code	sample mass	residue mass	melamine	other	d
	g	g	%	%	
136-1	0.8907	0.8336	93.98	6.02	1.068
136-2	0.7765	0.7270	94.02	5.98	1.068
136-3	0.7613	0.7149	94.28	5.72	1.065
136-4	0.8402	0.7922	94.64	5.36	1.061
136-5	0.7037	0.6606	94.25	5.75	1.065
136-6	0.8455	0.7998	94.93	5.07	1.057
136-7	0.9035	0.8456	93.99	6.01	1.068
136-8	0.8432	0.7888	93.94	6.06	1.069
136-9	1.0516	0.9947	94.93	5.07	1.057
136-10	1.0431	0.9760	93.96	6.04	1.069
136-11	1.0178	0.9521	93.94	6.06	1.069
average			**94.29**	**5.71**	**1.065**
uncertainty			**0.27**	**0.27**	**0.003**
SD			**0.40**	**0.40**	**0.005**
RSD%			**0.42**	**6.97**	**0.45**

100% cotton (sample 005)

JRC code	sample mass	residue mass	cotton	other
	g	g	%	%
005-6	0.8161	0.0006	99.93	0.07
005-7	0.8478	0.0003	99.97	0.03
005-8	0.7550	0.0004	99.95	0.05
005-9	0.8517	0.0003	99.97	0.03
005-10	0.8459	0.0002	99.98	0.02
average			99.96	0.04
uncertainty			0.02	0.02
SD			0.02	0.02
RSD%			0.02	44.12

100% viscose (sample034)

JRC code	sample mass	residue mass	other	viscose
	g	g	%	%
034-1	0.7300	0.0007	0.08	99.92
034-2	0.9530	0.0005	0.05	99.95
034-3	0.9169	0.0008	0.08	99.92
034-4	1.1151	0.0009	0.07	99.93
034-5	0.7424	0.0005	0.06	99.94
034-6	0.7746	0.0006	0.07	99.93
034-7	0.7577	0.0003	0.04	99.96
034-8	0.6932	0.0005	0.06	99.94
034-9	0.7070	0.0004	0.05	99.95
034-10	0.7655	0.0004	0.05	99.95
average			0.06	99.94
uncertainty			0.01	0.01
SD			0.02	0.02
RSD%			26.08	0.02

100% lyocell (sample 152)

JRC code	sample mass	residue mass	other	lyocell
	g	g	%	%
152-1	1.0529	0.0006	0.05	99.95
152-2	0.9060	0.0005	0.05	99.95
152-3	0.8435	0.0004	0.04	99.96
152-4	0.7376	0.0004	0.05	99.95
152-5	0.8463	0.0002	0.02	99.98
152-6	0.9929	0.0009	0.08	99.92
152-7	1.0148	0.0003	0.03	99.97
152-8	0.9798	0.0004	0.04	99.96
152-9	0.9947	0.0005	0.04	99.96
152-10	0.9967	0.0006	0.05	99.95
average			0.05	99.95
uncertainty			0.01	0.01
SD			0.02	0.02
RSD%			36.14	0.02

100% polyester (sample 153)

JRC code	sample mass	residue mass	polyester	other	d
	g	g	%	%	
153-1	1.0954	1.0670	97.44	2.56	1.027
153-2	1.0517	1.0226	97.27	2.73	1.028
153-3	0.9964	0.9711	97.50	2.50	1.026
153-4	1.0474	1.0185	97.28	2.72	1.028
153-5	1.0264	0.9990	97.37	2.63	1.027
153-6	1.0717	1.0434	97.40	2.60	1.027
153-7	1.0727	1.0447	97.43	2.57	1.027
153-8	1.0783	1.0504	97.45	2.55	1.027
153-9	1.0360	1.0082	97.36	2.64	1.028
153-10	1.0829	1.0531	97.29	2.71	1.028
average			**97.38**	**2.62**	**1.027**
uncertainty			**0.06**	**0.06**	**0.001**
SD			**0.08**	**0.08**	**0.001**
RSD%			**0.08**	**3.01**	**0.08**

100% modacrylic (sample 154)

JRC code	sample mass	residue mass	modacrylic	other	d
	g	g	%	%	
154-1	1.0423	1.0302	98.86	1.14	1.012
154-2	1.0679	1.0560	98.91	1.09	1.011
154-3	1.0078	0.9948	98.74	1.26	1.013
154-4	1.0158	1.0039	98.85	1.15	1.012
154-5	0.9965	0.9856	98.93	1.07	1.011
154-6	1.0560	1.0444	98.92	1.08	1.011
154-7	1.0599	1.0468	98.79	1.21	1.013
154-8	1.0441	1.0335	99.00	1.00	1.010
154-9	1.0633	1.0497	98.75	1.25	1.013
154-10	1.0321	1.0188	98.74	1.26	1.013
average			**98.85**	**1.15**	**1.012**
uncertainty			**0.07**	**0.07**	**0.001**
SD			**0.09**	**0.09**	**0.001**
RSD%			**0.10**	**8.15**	**0.10**

Chemical quantification

40% melamine – 60% m-aramid (Nomex) (sample 138)

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
50% H2SO4	138-1	1.0670	0.6699	63.63	36.37
90°C	138-2	1.0107	0.6349	63.66	36.34
1 hour	138-3	0.9665	0.6186	64.86	35.14
	138-4	0.9268	0.5868	64.16	35.84
	138-5	1.0054	0.6314	63.64	36.36
	138-6	1.0294	0.6483	63.82	36.18
	138-7	1.0473	0.6563	63.51	36.49
	138-8	1.0344	0.6511	63.79	36.21
	138-9	1.0309	0.6534	64.23	35.77
	138-10	0.9323	0.5896	64.09	35.91
	average			63.94	36.06
	uncertainty			0.29	0.29
	SD			0.41	0.41
	RSD%			0.63	1.12

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
50% H2SO4	138-1	0.9757	0.6199	64.38	35.62
90°C	138-2	0.8686	0.5469	63.81	36.19
3 hours	138-3	0.9976	0.6338	64.38	35.62
	138-4	1.0359	0.6591	64.48	35.52
	138-5	0.9406	0.5909	63.67	36.33
	138-6	0.9439	0.5971	64.11	35.89
	138-7	0.9667	0.6160	64.57	35.43
	138-8	0.9421	0.5968	64.20	35.80
	138-9	1.0291	0.6487	63.88	36.12
	138-10	1.1154	0.7007	63.66	36.34
	average			64.11	35.89
	uncertainty			0.24	0.24
	SD			0.34	0.34
	RSD%			0.53	0.95

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	138-1	1.0754	0.6361	59.97	40.03
90°C	138-2	0.9214	0.5467	60.15	39.85
1 hour	138-3	1.0703	0.6339	60.04	39.96
	138-4	1.0772	0.6435	60.56	39.44
	138-5	1.0415	0.6153	59.89	40.11
	138-6	0.9567	0.5785	61.29	38.71
	138-7	1.0995	0.6499	59.92	40.08
	138-8	0.9645	0.5740	60.33	39.67
	138-9	1.1087	0.6634	60.66	39.34
	138-10	1.0775	0.6429	60.49	39.51
	average			60.33	39.67
	uncertainty			0.31	0.31
	SD			0.44	0.44
	RSD%			0.72	1.10

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	138-1	0.9667	0.5755	60.35	39.65
90°C	138-2	1.0520	0.6271	60.43	39.57
3 hours	138-3	0.8920	0.5315	60.40	39.60
	138-4	1.0204	0.6146	61.06	38.94
	138-5	1.0236	0.6151	60.91	39.09
	138-6	0.8482	0.5064	60.52	39.48
	138-7	0.8056	0.4771	60.04	39.96
	138-8	0.9895	0.5819	59.62	40.38
	138-9	0.8873	0.5263	60.13	39.87
	138-10	0.8406	0.4968	59.92	40.08
	average			60.34	39.66
	uncertainty			0.31	0.31
	SD			0.44	0.44
	RSD%			0.72	1.10

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
method 14	M14-138-1	0.9843	0.4531	53.74	46.26
	M14-138-2	0.9659	0.4329	54.96	45.04
	M14-138-3	1.0329	0.4723	54.05	45.95
	M14-138-4	0.9350	0.4203	54.83	45.17
	M14-138-5	1.0484	0.4739	54.58	45.42
	M14-138-6	0.9852	0.4576	53.32	46.68
	M14-138-7	0.9748	0.4444	54.19	45.81
	M14-138-8	0.9898	0.4570	53.60	46.40
	M14-138-9	0.8670	0.4048	53.08	46.92
	M14-138-10	1.0053	0.4713	52.88	47.12
	average			53.92	46.08
	uncertainty			0.52	0.52
	SD			0.72	0.72
	RSD%			1.34	1.57

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
method 14	M14-138-1	0.7768	0.2978	61.50	38.50
liquid	M14-138-2	1.3327	0.5119	61.43	38.57
nitrogen	M14-138-3	0.7573	0.2933	61.10	38.90
	M14-138-4	0.8044	0.3156	60.60	39.40
	M14-138-5	0.9650	0.3767	60.80	39.20
	M14-138-6	0.8308	0.3300	60.11	39.89
	M14-138-7	0.8127	0.3205	60.39	39.61
	M14-138-8	1.0831	0.4211	60.95	39.05
	M14-138-9	1.1334	0.4437	60.68	39.32
	M14-138-10	1.3035	0.5155	60.28	39.72
	average			60.78	39.22
	uncertainty			0.33	0.33
	SD			0.47	0.47
	RSD%			0.77	1.19

40% melamine – 60% m-aramid (Nomex) (sample 139)

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
50% H2SO4	139-1	1.0497	0.6974	67.31	32.69
90°C	139-2	1.0756	0.7110	66.97	33.03
1 hour	139-3	0.9755	0.6496	67.46	32.54
	139-4	1.0467	0.6968	67.44	32.56
	139-5	0.9771	0.6490	67.29	32.71
	139-6	1.0229	0.6742	66.78	33.22
	139-7	1.0474	0.6899	66.73	33.27
	139-8	1.0050	0.6680	67.34	32.66
	139-9	1.0347	0.6872	67.28	32.72
	139-10	1.0683	0.7008	66.46	33.54
	average			67.11	32.89
	uncertainty			0.25	0.25
	SD			0.35	0.35
	RSD%			0.52	1.05

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
50% H2SO4	139-1	1.0538	0.7353	70.67	29.33
90°C	139-2	1.0156	0.7045	70.26	29.74
3 hours	139-3	1.0502	0.7285	70.26	29.74
	139-4	1.1382	0.7922	70.49	29.51
	139-5	0.9988	0.7022	71.20	28.80
	139-6	0.9659	0.6804	71.34	28.66
	139-7	0.9419	0.6558	70.52	29.48
	139-8	0.8988	0.6245	70.37	29.63
	139-9	0.9552	0.6688	70.91	29.09
	139-10	0.7632	0.5305	70.40	29.60
	average			70.64	29.36
	uncertainty			0.28	0.28
	SD			0.38	0.38
	RSD%			0.54	1.31

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	139-1	0.9954	0.6431	65.46	34.54
90°C	139-2	1.0123	0.6540	65.46	34.54
1 hour	139-3	0.9515	0.6155	65.54	34.46
	139-4	0.9980	0.6437	65.35	34.65
	139-5	0.9624	0.6242	65.72	34.28
	139-6	0.9441	0.6142	65.92	34.08
	139-7	0.9649	0.6273	65.87	34.13
	139-8	0.9795	0.6346	65.65	34.35
	139-9	0.9893	0.6391	65.46	34.54
	139-10	0.9731	0.6321	65.82	34.18
	139-11	1.3164	0.8457	65.10	34.90
	139-12	1.1882	0.7607	64.87	35.13
	139-13	1.4975	0.9623	65.11	34.89
	139-14	1.0978	0.7010	64.71	35.29
	139-15	1.1422	0.7220	64.06	35.94
	average			65.34	34.66
	uncertainty			0.28	0.28
	SD			0.50	0.50
	RSD%			0.77	1.45

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	139-1	0.9044	0.5648	63.29	36.71
90°C	139-2	1.0094	0.6231	62.57	37.43
3 hours	139-3	1.0075	0.6295	63.32	36.68
	139-4	1.1013	0.6816	62.73	37.27
	139-5	1.0414	0.6474	63.01	36.99
	139-6	1.0543	0.6834	65.68	34.32
	139-7	0.9128	0.5991	66.50	33.50
	139-8	1.0062	0.6542	65.88	34.12
	139-9	1.0531	0.6844	65.85	34.15
	139-10	0.9560	0.6247	66.21	33.79
	139-11	1.0057	0.6465	65.14	34.86
	139-12	1.0162	0.6508	64.90	35.10
	139-13	1.0613	0.6805	64.97	35.03
	139-14	1.0030	0.6456	65.22	34.78
	139-15	0.9952	0.6397	65.13	34.87
	139-16	0.9984	0.6398	64.94	35.06
	139-17	0.9838	0.6312	65.01	34.99
	139-18	0.9544	0.6126	65.04	34.96
	139-19	1.0156	0.6514	64.99	35.01
	139-20	0.9817	0.6324	65.27	34.73
	average			64.78	35.22
	uncertainty			0.54	0.54
	SD			1.16	1.16
	RSD%			1.79	3.30

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
method 14	M14-139-1	1.0519	0.2890	72.44	27.56
liquid	M14-139-2	1.0230	0.3310	67.52	32.48
nitrogen	M14-139-3	0.9933	0.3096	68.72	31.28
	M14-139-4	1.0606	0.3384	67.98	32.02
	M14-139-5	0.8150	0.2486	69.39	30.61
	M14-139-6	1.0290	0.3065	70.11	29.89
	M14-139-7	1.1943	0.3355	71.82	28.18
	M14-139-8	1.0216	0.3277	67.81	32.19
	M14-139-9	0.9126	0.3317	63.51	36.49
	M14-139-10	0.9362	0.2261	75.78	24.22
	average			69.51	30.49
	uncertainty			2.38	2.38
	SD			3.32	3.32
	RSD%			4.78	10.90

50% melamine – 50% m-aramid (Nomex) (sample 140)

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
50% H2SO4	140-1	0.9587	0.4915	52.01	47.99
90°C	140-2	0.9895	0.5088	52.17	47.83
1 hour	140-3	1.0097	0.5264	52.89	47.11
	140-4	0.9766	0.4984	51.78	48.22
	140-5	1.0134	0.5229	52.35	47.65
	140-6	0.9589	0.4967	52.55	47.45
	140-7	1.0232	0.5234	51.90	48.10
	140-8	1.0436	0.5355	52.06	47.94
	140-9	0.9833	0.5077	52.38	47.62
	140-10	1.0179	0.5278	52.60	47.40
	average			52.27	47.73
	uncertainty			0.25	0.25
	SD			0.35	0.35
	RSD%			0.67	0.73

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
50% H2SO4	140-1	1.0379	0.5255	51.37	48.63
90°C	140-2	0.9261	0.4722	51.73	48.27
3 hours	140-3	1.0337	0.5262	51.65	48.35
	140-4	1.0246	0.5214	51.63	48.37
	140-5	0.8515	0.4353	51.87	48.13
	140-6	1.0174	0.5208	51.93	48.07
	140-7	0.9901	0.5078	52.03	47.97
	140-8	0.9740	0.5006	52.14	47.86
	140-9	1.0377	0.5327	52.08	47.92
	140-10	0.9826	0.4999	51.62	48.38
	average			51.80	48.20
	uncertainty			0.18	0.18
	SD			0.25	0.25
	RSD%			0.48	0.51

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	140-1	1.0000	0.4999	50.72	49.28
90°C	140-2	0.9405	0.4769	51.45	48.55
1 hour	140-3	0.8745	0.4369	50.69	49.31
	140-4	0.9632	0.4848	51.07	48.93
	140-5	0.9485	0.4760	50.92	49.08
	140-6	1.0084	0.5026	50.57	49.43
	140-7	0.9916	0.4906	50.20	49.80
	140-8	0.9499	0.4702	50.23	49.77
	140-9	0.9544	0.4717	50.15	49.85
	140-10	1.0117	0.5058	50.73	49.27
	140-11	1.0011	0.5206	52.75	47.25
	140-12	0.9789	0.5067	52.51	47.49
	140-13	1.0033	0.5199	52.57	47.43
	140-14	0.9771	0.5045	52.38	47.62
	140-15	0.9846	0.5108	52.63	47.37
	140-16	0.9833	0.5055	52.15	47.85
	140-17	0.9722	0.4985	52.02	47.98
	140-18	0.9553	0.4905	52.09	47.91
	140-19	0.9696	0.4976	52.07	47.93
	140-20	1.0139	0.5176	51.79	48.21
	average			51.48	48.52
	uncertainty			0.42	0.42
	SD			0.91	0.91
	RSD%			1.76	1.87

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	140-1	0.7251	0.3693	51.67	48.33
90°C	140-2	0.9759	0.4947	51.43	48.57
3 hours	140-3	0.8336	0.4197	51.08	48.92
	140-4	0.7575	0.3847	51.53	48.47
	140-5	1.0109	0.5110	51.29	48.71
	140-6	1.0099	0.5146	51.70	48.30
	140-7	1.0568	0.5332	51.19	48.81
	140-8	1.0124	0.5108	51.19	48.81
	140-9	0.9975	0.5050	51.37	48.63
	140-10	0.9989	0.5037	51.16	48.84
	average			51.36	48.64
	uncertainty			0.15	0.15
	SD			0.22	0.22
	RSD%			0.42	0.45

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
method 14	M14-140-1	0.9215	0.4654	49.22	50.78
	M14-140-2	0.9683	0.4793	50.24	49.76
	M14-140-3	0.9245	0.4728	48.58	51.42
	M14-140-4	0.9686	0.4963	48.48	51.52
	M14-140-5	1.0062	0.5235	47.68	52.32
	M14-140-6	0.9792	0.4976	48.91	51.09
	M14-140-7	0.9970	0.5050	49.07	50.93
	M14-140-8	0.7098	0.3680	47.87	52.13
	M14-140-9	1.0763	0.5508	48.55	51.45
	M14-140-10	0.9869	0.5091	48.13	51.87
	average			48.67	51.33
	uncertainty			0.53	0.53
	SD			0.74	0.74
	RSD%			1.52	1.44

40% melamine – 60% p-aramid (Kevlar) (sample 141)

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
50% H2SO4	141-1	0.9968	0.6070	61.72	38.28
90°C	141-2	0.9759	0.6156	63.93	36.07
1 hour	141-3	0.9927	0.6136	62.65	37.35
	141-4	0.9406	0.5810	62.60	37.40
	141-5	0.9695	0.5946	62.16	37.84
	141-6	1.0621	0.6579	62.78	37.22
	141-7	1.0177	0.6176	61.51	38.49
	141-8	1.0780	0.6663	62.64	37.36
	141-9	0.9582	0.5903	62.44	37.56
	141-10	0.9905	0.6060	62.01	37.99
	average			62.45	37.55
	uncertainty			0.48	0.48
	SD			0.67	0.67
	RSD%			1.08	1.79

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
50% H2SO4	141-1	0.7160	0.4327	61.26	38.74
90°C	141-2	1.0480	0.6362	61.53	38.47
3 hours	141-3	1.0013	0.6052	61.27	38.73
	141-4	1.0084	0.6111	61.43	38.57
	141-5	1.0035	0.6114	61.76	38.24
	141-6	0.9416	0.5717	61.54	38.46
	141-7	0.9792	0.5965	61.75	38.25
	141-8	1.0743	0.6511	61.43	38.57
	141-9	0.9780	0.5919	61.35	38.65
	141-10	1.0113	0.6120	61.34	38.66
	average			61.47	38.53
	uncertainty			0.13	0.13
	SD			0.18	0.18
	RSD%			0.29	0.46

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
90% HCOOH	141-1	0.9669	0.5893	61.78	38.22
90°C	141-2	0.9923	0.6048	61.78	38.22
1 hour	141-3	0.9916	0.6008	61.42	38.58
	141-4	0.9954	0.6037	61.48	38.52
	141-5	0.9548	0.5832	61.91	38.09
	141-6	0.9987	0.6051	61.42	38.58
	141-7	1.0233	0.6186	61.28	38.72
	141-8	1.0001	0.6065	61.47	38.53
	141-9	0.9807	0.5936	61.35	38.65
	141-10	1.0478	0.6365	61.57	38.43
	average			61.54	38.46
	uncertainty			0.15	0.15
	SD			0.21	0.21
	RSD%			0.34	0.55

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
90% HCOOH	141-1	0.9622	0.5868	61.81	38.19
90°C	141-2	0.9553	0.5804	61.58	38.42
3 hours	141-3	0.9226	0.5612	61.66	38.34
	141-4	1.0074	0.6138	61.76	38.24
	141-5	0.9544	0.5815	61.76	38.24
	141-6	0.9879	0.5997	61.53	38.47
	141-7	0.9765	0.5925	61.50	38.50
	141-8	1.0104	0.6153	61.73	38.27
	141-9	0.9793	0.5972	61.81	38.19
	141-10	0.9811	0.5967	61.65	38.35
	average			61.68	38.32
	uncertainty			0.08	0.08
	SD			0.11	0.11
	RSD%			0.18	0.29

40% melamine – 60% p-aramid (Kevlar) (sample 142)

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
50% H2SO4	142-1	1.0134	0.6368	63.68	36.32
90°C	142-2	0.9381	0.6071	65.57	34.43
1 hour	142-3	1.0404	0.6643	64.70	35.30
	142-4	0.9518	0.6035	64.25	35.75
	142-5	0.9508	0.6021	64.17	35.83
	142-6	0.9258	0.5902	64.60	35.40
	142-7	1.0962	0.6941	64.17	35.83
	142-8	1.0698	0.6791	64.33	35.67
	142-9	1.0506	0.6662	64.26	35.74
	142-10	0.9322	0.5957	64.75	35.25
	average			64.45	35.55
	uncertainty			0.36	0.36
	SD			0.50	0.50
	RSD%			0.78	1.42

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
50% H2SO4	142-1	1.0505	0.6657	64.22	35.78
90°C	142-2	0.9788	0.6258	64.79	35.21
3 hours	142-3	0.9734	0.6215	64.70	35.30
	142-4	0.9741	0.6166	64.15	35.85
	142-5	0.9576	0.6075	64.29	35.71
	142-6	1.0128	0.6395	63.99	36.01
	142-7	0.9978	0.6359	64.58	35.42
	142-8	0.9896	0.6261	64.11	35.89
	142-9	1.0131	0.6435	64.37	35.63
	142-10	0.9594	0.6096	64.39	35.61
	average			64.36	35.64
	uncertainty			0.19	0.19
	SD			0.26	0.26
	RSD%			0.41	0.73

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
90% HCOOH	142-1	1.1388	0.6748	60.07	39.93
90°C	142-2	1.0302	0.6133	60.35	39.65
1 hour	142-3	0.9707	0.5779	60.35	39.65
	142-4	0.9684	0.5725	59.93	40.07
	142-5	1.0012	0.5913	59.87	40.13
	142-6	0.9143	0.5452	60.45	39.55
	142-7	1.0166	0.6061	60.44	39.56
	142-8	0.9527	0.5635	59.96	40.04
	142-9	1.0395	0.6164	60.11	39.89
	142-10	0.9839	0.5824	60.01	39.99
	average			60.16	39.84
	uncertainty			0.16	0.16
	SD			0.22	0.22
	RSD%			0.37	0.56

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
90% HCOOH	142-1	0.8585	0.5099	60.21	39.79
90°C	142-2	0.8670	0.5139	60.09	39.91
3 hours	142-3	0.9359	0.5563	60.26	39.74
	142-4	0.8144	0.4859	60.48	39.52
	142-5	0.8881	0.5276	60.22	39.78
	142-6	0.8895	0.5294	60.33	39.67
	142-7	0.8942	0.5311	60.21	39.79
	142-8	0.8365	0.5001	60.60	39.40
	142-9	0.8869	0.5277	60.32	39.68
	142-10	0.8910	0.5284	60.12	39.88
	average			60.29	39.71
	uncertainty			0.11	0.11
	SD			0.16	0.16
	RSD%			0.26	0.40

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
method 14	M14-142-1	0.7744	0.6357	17.22	82.78
	M14-142-2	0.9347	0.7801	15.83	84.17
	M14-142-3	0.7342	0.6083	16.45	83.55
	M14-142-4	0.8952	0.7338	17.34	82.66
	M14-142-5	0.8869	0.7338	16.56	83.44
	M14-142-6	1.0217	0.8378	17.31	82.69
	M14-142-7	0.9260	0.7674	16.43	83.57
	M14-142-8	0.8708	0.7152	17.18	82.82
	M14-142-9	0.8803	0.7217	17.33	82.67
	M14-142-10	0.9876	0.8122	17.07	82.93
	average			16.87	83.13
	uncertainty			0.37	0.37
	SD			0.52	0.52
	RSD%			3.09	0.63

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
method 14	M14-142-1	0.6808	0.5075	24.88	75.12
manual	M14-142-2	0.5880	0.4262	26.98	73.02
separation	M14-142-3	1.0027	0.7263	27.02	72.98
liquid	M14-142-4	1.0600	0.7683	26.98	73.02
nitrogen	M14-142-5	1.1739	0.8529	26.80	73.20
	M14-142-6	1.1027	0.7971	27.17	72.83
	M14-142-7	1.0270	0.7403	27.38	72.62
	M14-142-8	0.9397	0.6773	27.39	72.61
	M14-142-9	0.9558	0.6952	26.72	73.28
	M14-142-10	0.9497	0.6863	27.20	72.80
	average			26.85	73.15
	uncertainty			0.52	0.52
	SD			0.73	0.73
	RSD%			2.70	0.99

25% melamine – 75% m-aramid (Conex) (sample 143)

method	JRC code	sample mass g	residue mass g	m-aramid %	melamine %
50% H2SO4	143-1	0.9174	0.7653	84.38	15.62
90°C	143-2	1.0082	0.8442	84.69	15.31
1 hour	143-3	0.9295	0.7818	85.07	14.93
	143-4	0.9626	0.8088	84.98	15.02
	143-5	0.9286	0.7799	84.95	15.05
	143-6	1.0042	0.8380	84.41	15.59
	143-7	1.0027	0.8370	84.43	15.57
	143-8	0.8664	0.7257	84.72	15.28
	143-9	0.7132	0.5976	84.75	15.25
	143-10	0.6788	0.5659	84.32	15.68
	average			84.67	15.33
	uncertainty			0.20	0.20
	SD			0.27	0.27
	RSD%			0.32	1.78

method	JRC code	sample mass g	residue mass g	m-aramid %	melamine %
50% H2SO4	143-1	1.0078	0.8415	84.46	15.54
90°C	143-2	1.0452	0.8827	85.41	14.59
3 hours	143-3	0.9519	0.8055	85.58	14.42
	143-4	1.0211	0.8506	84.26	15.74
	143-5	0.9652	0.8109	84.97	15.03
	143-6	0.9368	0.7871	84.98	15.02
	143-7	1.0855	0.9131	85.08	14.92
	143-8	0.9461	0.7915	84.62	15.38
	143-9	0.8109	0.6821	85.08	14.92
	143-10	0.9150	0.7666	84.74	15.26
	average			84.92	15.08
	uncertainty			0.29	0.29
	SD			0.41	0.41
	RSD%			0.48	2.72

method	JRC code	sample mass g	residue mass g	m-aramid %	melamine %
90% HCOOH	143-1	0.9944	0.8032	81.72	18.28
90°C	143-2	1.0303	0.8278	81.29	18.71
1 hour	143-3	0.9327	0.7483	81.17	18.83
	143-4	0.9635	0.7779	81.68	18.32
	143-5	0.9854	0.7904	81.16	18.84
	143-6	0.9574	0.7668	81.04	18.96
	143-7	0.9582	0.7729	81.61	18.39
	143-8	0.9558	0.7694	81.44	18.56
	143-9	1.0029	0.8081	81.52	18.48
	143-10	0.9869	0.7927	81.27	18.73
	average			81.39	18.61
	uncertainty			0.17	0.17
	SD			0.24	0.24
	RSD%			0.29	1.28

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	143-1	0.9617	0.7715	81.17	18.83
90°C	143-2	0.6827	0.5452	80.80	19.20
3 hours	143-3	0.9679	0.7782	81.35	18.65
	143-4	0.9509	0.7680	81.71	18.29
	143-5	0.9505	0.7703	81.99	18.01
	143-6	1.0020	0.8083	81.62	18.38
	143-7	1.0108	0.8138	81.46	18.54
	143-8	1.1018	0.8817	80.97	19.03
	143-9	1.0301	0.8264	81.17	18.83
	143-10	1.0099	0.8060	80.75	19.25
	average			81.30	18.70
	uncertainty			0.29	0.29
	SD			0.40	0.40
	RSD%			0.50	2.16

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
90% HCOOH	143-1	1.0582	0.2132	79.80	20.20
90°C	143-2	1.0534	0.2221	78.86	21.14
3 hours	143-3	0.9203	0.1723	81.23	18.77
liquid	143-4	0.9649	0.1729	82.04	17.96
nitrogen	143-5	0.9993	0.1834	81.60	18.40
	143-6	1.1932	0.2037	82.89	17.11
	143-7	1.1668	0.2105	81.92	18.08
	143-8	1.4112	0.2659	81.11	18.89
	average			81.18	18.82
	uncertainty			1.08	1.08
	SD			1.29	1.29
	RSD%			1.59	6.86

17% melamine – 17% modacrylic – 66% polyester (sample 144)

method	JRC code	sample mass	residue PES+mod	residue PES	melamine	PES	modacrylic
		g	g	g	%	%	%
VARIANT 4	144-1	0.9017	0.7552	0.6045	16.96	67.07	16.04
50% H2SO4	144-2	0.9194	0.7701	0.6187	16.96	67.32	15.79
90°C	144-3	1.0276	0.8599	0.6942	17.04	67.58	15.45
1 hour	144-4	1.1566	0.9707	0.7753	16.78	67.07	16.22
method 8	144-5	0.8800	0.7331	0.5835	17.43	66.32	16.34
	144-6	1.0608	0.8870	0.7085	17.11	66.81	16.16
	144-7	1.0868	0.9063	0.7297	17.34	67.16	15.58
	144-8	1.0328	0.8652	0.6822	16.94	66.08	17.06
	144-9	1.0620	0.8851	0.7151	17.39	67.35	15.33
	144-10	1.0405	0.8719	0.6986	16.92	67.17	15.98
	average			0.68	17.09	66.99	16.00
	uncertainty			0.04	0.16	0.34	0.36
	SD			0.06	0.22	0.47	0.50
	RSD%			8.88	1.30	0.70	3.14

method	JRC code	sample mass	residue PES+mod	residue PES	melamine	PES	modacrylic
		g	g	g	%	%	%
VARIANT 4	144-1	0.9927	0.8282	0.6559	17.30	66.09	16.70
50% H2SO4	144-2	1.2656	1.0588	0.8383	17.06	66.26	16.76
90°C	144-3	1.1709	0.9764	0.7722	17.34	65.96	16.78
3 hours	144-4	0.9758	0.8161	0.6428	17.09	65.89	17.10
method 8	144-5	0.9730	0.8129	0.6422	17.18	66.02	16.88
	average			0.71	17.19	66.04	16.84
	uncertainty			0.11	0.15	0.17	0.20
	SD			0.09	0.12	0.14	0.16
	RSD%			12.66	0.73	0.21	0.94

method	JRC code	sample mass	residue PES+melamine	modacrylic
		g	g	%
VARIANT 2	144-1	1.0467	0.8703	15.93
method 8	144-2	1.0762	0.8919	16.21
modacrylic	144-3	0.9389	0.7830	15.68
soluble	144-4	0.9304	0.7699	16.33
	144-5	0.9452	0.7835	16.19
	144-6	0.8767	0.7252	16.37
	144-7	0.9961	0.8237	16.39
	144-8	1.0617	0.8745	16.72
	144-9	0.9912	0.8175	16.61
	144-10	1.1010	0.9073	16.68
	144-11	1.1123	0.9213	16.26
	144-12	0.7924	0.6575	16.11
	144-13	1.0637	0.8815	16.22
	144-14	1.0805	0.8986	15.92
	144-15	0.9626	0.7961	16.39
	average		0.83	16.27
	uncertainty		0.04	0.16
	SD		0.08	0.29
	RSD%		9.09	1.75

method	JRC code	sample mass g	residue melamine g	melamine %
VARIANT 2	144-1	0.9810	0.1742	18.71
method 14	144-2	0.9451	0.1670	18.62
modacrylic,	144-3	0.9640	0.1703	18.62
PES soluble	144-4	0.8870	0.1574	18.70
	144-5	0.9894	0.1749	18.63
	144-6	0.9964	0.1730	18.30
	144-7	1.0333	0.1810	18.46
	144-8	1.0530	0.1831	18.32
	144-9	0.9652	0.1710	18.67
	144-10	0.9897	0.1731	18.43
	144-11	1.0488	0.1768	17.77
	144-12	1.1888	0.2007	17.80
	144-13	1.1121	0.1889	17.90
	144-14	1.1306	0.1918	17.88
	144-15	1.2270	0.1995	17.15
		average		18.26
		uncertainty		0.26
		SD		0.46
		RSD%		2.53

12% melamine – 24% lm polyester – 30% lyocell - 34% modacrylic (sample 145)

method	JRC code	sample mass	PES+mod+mel	PES+mel	lyocell	PES	modacrylic	melamine
		g	g	g	%	%	%	%
VARIANT 4	145-1	1.0196	0.7108	0.3637	32.71	22.74	32.23	12.32
method 7	145-2	0.9453	0.6484	0.3266	33.84	20.71	32.20	13.25
method 8	145-3	0.9554	0.6658	0.3387	32.72	21.38	32.41	13.50
method 14	145-4	0.9343	0.6618	0.3304	31.55	22.73	33.65	12.07
	145-5	1.0181	0.7040	0.3575	33.27	21.33	32.21	13.19
	145-6	1.1000	0.7689	0.3970	32.51	22.50	32.00	12.99
	145-7	1.0428	0.7304	0.3889	32.37	23.23	30.96	13.44
	145-8	1.0105	0.7265	0.3661	30.44	22.25	33.85	13.46
	145-9	1.0441	0.7361	0.3748	31.89	22.60	32.79	12.72
	145-10	0.9713	0.6919	0.3468	31.14	22.91	33.71	12.23
	average				32.25	22.24	32.60	12.92
	uncertainty				0.72	0.59	0.65	0.39
	SD				1.01	0.82	0.91	0.55
	RSD%				3.14	3.69	2.80	4.23

method	JRC code	sample mass	residue melamine	melamine	lyocell	PES	modacrylic
		g	g	%	%	%	%
VARIANT 2	145-1	0.8951	0.1059	12.25	30.54	24.16	33.04
method 14	145-2	0.9257	0.1101	12.31	30.56	23.33	33.81
modacrylic,	145-3	0.9331	0.1110	12.31	30.70	23.10	33.89
lyocell,	145-4	0.9541	0.1125	12.21	30.35	24.20	33.24
PES soluble	145-5	0.9571	0.1120	12.12	30.44	24.25	33.19
	145-6	0.9909	0.1158	12.10	30.42	24.28	33.20
	145-7	1.0130	0.1199	12.26	29.96	24.68	33.10
	145-8	0.9908	0.1177	12.30	30.62	23.55	33.53
	145-9	1.1074	0.1309	12.24	30.67	23.68	33.40
	145-10	1.0969	0.1290	12.18	30.54	23.60	33.68
	average		0.12	12.23	30.48	23.88	33.41
	uncertainty		0.01	0.05	0.15	0.36	0.22
	SD		0.01	0.08	0.21	0.50	0.30
	RSD%		6.99	0.62	0.70	2.10	0.91

melamine-viscose (sample 172)

method	JRC code	sample mass	residue mass	viscose	melamine
		g	g	%	%
method 3	M3-172-1	0.8197	0.4190	50.25	49.75
	M3-172-2	0.8077	0.4155	49.92	50.08
	M3-172-3	0.7340	0.3817	49.36	50.64
	M3-172-4	0.8755	0.4416	50.92	49.08
	M3-172-5	0.8318	0.4212	50.73	49.27
	M3-172-6	0.9964	0.5081	50.37	49.63
	M3-172-7	0.9653	0.4917	50.43	49.57
	M3-172-8	0.9801	0.5007	50.28	49.72
	M3-172-9	1.0799	0.5463	50.78	49.22
	M3-172-10	1.0010	0.5111	50.30	49.70
	average			50.33	49.67
	uncertainty			0.32	0.32
	SD			0.45	0.45
	RSD%			0.90	0.91

method	JRC code	sample mass	residue mass	viscose	melamine
		g	g	%	%
method 7	M7-172-1	0.9837	0.4863	51.43	48.57
	M7-172-2	0.9377	0.4633	51.46	48.54
	M7-172-3	0.9986	0.4949	51.31	48.69
	M7-172-4	1.0213	0.5028	51.64	48.36
	M7-172-5	0.9949	0.5028	50.32	49.68
	M7-172-6	0.8065	0.4098	50.04	49.96
	M7-172-7	0.8297	0.4091	51.56	48.44
	M7-172-8	0.8153	0.4084	50.77	49.23
	M7-172-9	0.7846	0.3945	50.58	49.42
	M7-172-10	0.7716	0.3866	50.76	49.24
	M7-172-11	0.8465	0.4302	50.03	49.97
	M7-172-12	0.8171	0.4107	50.60	49.40
	M7-172-13	1.0047	0.5054	50.56	49.44
	M7-172-14	1.0107	0.5092	50.48	49.52
	M7-172-15	0.9820	0.4952	50.43	49.57
		average		50.80	49.20
		uncertainty		0.30	0.30
		SD		0.55	0.55
		RSD%		1.07	1.11

method	JRC code	sample mass	residue mass	viscose	melamine
		g	g	%	%
method 14	M14-172-1	1.0044	0.5023	50.85	49.15
	M14-172-2	0.9832	0.4982	50.19	49.81
	M14-172-3	1.0148	0.5169	49.92	50.08
	M14-172-4	1.0922	0.5479	50.70	49.30
	M14-172-5	1.1461	0.5825	50.03	49.97
	M14-172-6	1.0103	0.5103	50.35	49.65
	M14-172-7	1.0050	0.5107	50.04	49.96
	M14-172-8	1.0112	0.5088	50.54	49.46
	M14-172-9	0.9454	0.4721	50.93	49.07
	M14-172-10	1.0018	0.5213	48.81	51.19
	average			50.24	49.76
	uncertainty			0.44	0.44
	SD			0.62	0.62
	RSD%			1.23	1.24

melamine-Visil (sample 173)

method	JRC code	sample mass g	residue mass g	Visil %	melamine %
method 3	M3-173-1	0.7817	0.6693	15.06	84.94
	M3-173-2	0.9303	0.7957	15.16	84.84
	M3-173-3	0.8198	0.7010	15.18	84.82
	M3-173-4	0.8611	0.7370	15.10	84.90
	M3-173-5	0.8851	0.7579	15.06	84.94
	M3-173-6	0.9905	0.8482	15.05	84.95
	M3-173-7	1.0602	0.9099	14.85	85.15
	M3-173-8	1.0000	0.8560	15.09	84.91
	M3-173-9	1.0106	0.8709	14.49	85.51
	M3-173-10	1.0096	0.8669	14.81	85.19
	average			14.98	85.02
	uncertainty			0.15	0.15
	SD			0.21	0.21
	RSD%			1.41	0.25

method	JRC code	sample mass g	residue mass g	Visil %	melamine %
method 7	M7-173-1	0.8908	0.1877	79.62	20.38
	M7-173-2	0.7965	0.1651	79.95	20.05
	M7-173-3	0.9522	0.1997	79.71	20.29
	M7-173-4	0.7955	0.1687	79.49	20.51
	M7-173-5	0.9624	0.2014	79.76	20.24
	M7-173-6	1.0106	0.2081	80.09	19.91
	M7-173-7	1.0107	0.2111	79.80	20.20
	M7-173-8	1.0522	0.2199	79.79	20.21
	M7-173-9	1.0998	0.2302	79.75	20.25
	M7-173-10	1.0052	0.2082	79.97	20.03
	average			79.79	20.21
	uncertainty			0.13	0.13
	SD			0.18	0.18
	RSD%			0.22	0.87

method	JRC code	sample mass g	residue mass g	Visil %	melamine %
method 14	M14-173-1	0.8841	0.1814	80.16	19.84
	M14-173-2	0.8399	0.1732	80.06	19.94
	M14-173-3	0.8955	0.1865	79.86	20.14
	M14-173-4	0.7528	0.1590	79.57	20.43
	M14-173-5	0.8488	0.1775	79.77	20.23
	M14-173-6	1.0103	0.2098	79.92	20.08
	M14-173-7	0.9975	0.2078	79.85	20.15
	M14-173-8	1.0146	0.2091	80.07	19.93
	M14-173-9	1.0077	0.2089	79.95	20.05
	M14-173-10	1.0102	0.2100	79.89	20.11
	average			79.91	20.09
	uncertainty			0.12	0.12
	SD			0.17	0.17
	RSD%			0.21	0.83

melamine-polyester (sample 174)

method	JRC code	sample mass	residue mass	PES	melamine
		g	g	%	%
50% H2SO4	174-1	1.0085	0.6201	60.23	39.77
90°C	174-2	1.0124	0.6253	60.51	39.49
1 hour	174-3	0.9903	0.6128	60.63	39.37
	174-4	0.9893	0.6145	60.87	39.13
	174-5	1.0491	0.6482	60.53	39.47
	174-6	1.0050	0.6218	60.62	39.38
	174-7	1.0093	0.6220	60.37	39.63
	174-8	1.0100	0.6232	60.45	39.55
	174-9	1.0099	0.6238	60.51	39.49
	174-10	1.0020	0.6160	60.22	39.78
	average			60.49	39.51
	uncertainty			0.14	0.14
	SD			0.19	0.19
	RSD%			0.32	0.49

method	JRC code	sample mass	residue mass	PES	melamine
		g	g	%	%
method 14	M14-174-1	0.9953	0.3767	60.52	39.48
	M14-174-2	0.9818	0.3728	60.39	39.61
	M14-174-3	1.0407	0.3943	60.48	39.52
	M14-174-4	1.0019	0.3791	60.53	39.47
	M14-174-5	0.9612	0.3661	60.27	39.73
	M14-174-6	1.0101	0.3903	59.71	40.29
	M14-174-7	0.9772	0.3758	59.90	40.10
	M14-174-8	1.0113	0.3897	59.82	40.18
	M14-174-9	1.0390	0.3988	59.97	40.03
	M14-174-10	1.0004	0.3831	60.06	39.94
	average			60.17	39.83
	uncertainty			0.22	0.22
	SD			0.31	0.31
	RSD%			0.52	0.78

melamine-cotton (sample 175)

method	JRC code	sample mass	residue mass	Cotton	Melamine
		g	g	%	%
90 % HCOOH	175-1	0.9192	0.3819	41.89	58.11
90 °C	175-2	1.0310	0.4281	41.86	58.14
1 hour	175-3	1.0699	0.4423	41.68	58.32
	175-4	0.9133	0.3726	41.13	58.87
	175-5	1.0264	0.4252	41.76	58.24
	175-6	1.0788	0.4472	41.79	58.21
	175-7	0.9845	0.4096	41.94	58.06
	175-8	1.0436	0.4284	41.39	58.61
	175-9	1.0116	0.4191	41.77	58.23
	175-10	0.9903	0.4052	41.25	58.75
	average			41.65	58.35
	uncertainty			0.20	0.20
	SD			0.28	0.28
	RSD%			0.68	0.49

method	JRC code	sample mass	residue mass	Cotton	Melamine
		g	g	%	%
90 % HCOOH	175-1	0.9807	0.3829	39.38	60.62
90 °C	175-2	0.9325	0.3696	39.97	60.03
1 hour	175-3	1.1505	0.4542	39.81	60.19
	175-4	0.8508	0.3373	39.98	60.02
manual	175-5	0.8580	0.3372	39.63	60.37
separation	175-6	1.0080	0.3952	39.54	60.46
before	175-7	1.0137	0.4007	39.86	60.14
chemical	175-8	0.9900	0.3885	39.57	60.43
analysis	175-9	0.9202	0.3632	39.80	60.20
	175-10	0.8944	0.3484	39.29	60.71
	average			39.68	60.32
	uncertainty			0.17	0.17
	SD			0.24	0.24
	RSD%			0.61	0.40

method	JRC code	sample mass	residue mass	Cotton	Melamine
		g	g	%	%
method 7	175-1	1.0167	0.6218	38.56	61.44
	175-2	1.0025	0.6116	38.71	61.29
	175-3	1.0530	0.6426	38.69	61.31
	175-4	1.0002	0.6125	38.48	61.52
	175-5	0.9754	0.5979	38.42	61.58
	175-6	1.0215	0.6124	39.78	60.22
	175-7	0.9349	0.5630	39.51	60.49
	175-8	1.0151	0.6084	39.80	60.20
	175-9	1.0085	0.6089	39.35	60.65
	175-10	1.0124	0.6097	39.51	60.49
	average			39.08	60.92
	uncertainty			0.40	0.40
	SD			0.56	0.56
	RSD%			1.43	0.92

method	JRC code	sample mass	residue mass	Cotton	Melamine
		g	g	%	%
method 14	**175-1**	1.0775	0.6610	38.37	61.63
	175-2	1.0300	0.6280	38.75	61.25
	175-3	0.9951	0.6100	38.42	61.58
	175-4	0.9538	0.5827	38.63	61.37
	175-5	1.0151	0.6160	39.04	60.96
	175-6	1.0180	0.6226	38.56	61.44
	175-7	1.0069	0.6162	38.52	61.48
	175-8	1.0122	0.6192	38.54	61.46
	175-9	1.0118	0.6153	38.91	61.09
	175-10	1.0088	0.6034	39.92	60.08
	average			**38.77**	**61.23**
	uncertainty			**0.33**	**0.33**
	SD			**0.46**	**0.46**
	RSD%			**1.18**	**0.75**

melamine-viscose (sample 176)

method	JRC code	sample mass	residue mass	viscose	melamine
		g	g	%	%
method 3	M3-176-1	0.8765	0.3764	58.39	41.61
	M3-176-2	0.9910	0.4233	58.61	41.39
	M3-176-3	0.8092	0.3441	58.80	41.20
	M3-176-4	1.0096	0.4314	58.60	41.40
	M3-176-5	1.0089	0.4302	58.69	41.31
	M3-176-6	1.0065	0.4345	58.16	41.84
	M3-176-7	1.1049	0.4728	58.54	41.46
	M3-176-8	1.0476	0.4487	58.50	41.50
	M3-176-9	0.9992	0.4235	58.94	41.06
	M3-176-10	1.0107	0.4338	58.41	41.59
	average			58.56	41.44
	uncertainty			0.14	0.14
	SD			0.22	0.22
	RSD%			0.38	0.53

method	JRC code	sample mass	residue mass	viscose	melamine
		g	g	%	%
method 7	M7-176-1	0.8628	0.3598	59.21	40.79
	M7-176-2	0.8144	0.3371	59.51	40.49
	M7-176-3	0.8731	0.3638	59.24	40.76
	M7-176-4	0.7705	0.3214	59.19	40.81
	M7-176-5	0.9075	0.3753	59.55	40.45
	M7-176-6	1.0110	0.4206	59.30	40.70
	M7-176-7	1.0127	0.4233	59.11	40.89
	M7-176-8	0.9112	0.3766	59.58	40.42
	M7-176-9	0.9992	0.4155	59.32	40.68
	M7-176-10	1.0070	0.4194	59.26	40.74
	average			59.33	40.67
	uncertainty			0.12	0.12
	SD			0.16	0.16
	RSD%			0.28	0.40

method	JRC code	sample mass	residue mass	viscose	melamine
		g	g	%	%
method 14	M14-176-1	0.7548	0.3170	58.91	41.09
	M14-176-2	0.7882	0.3271	59.41	40.59
	M14-176-3	0.7532	0.3138	59.24	40.76
	M14-176-4	0.6928	0.2842	59.89	40.11
	M14-176-5	0.8274	0.3456	59.14	40.86
	M14-176-6	1.0100	0.4231	59.02	40.98
	M14-176-7	1.0043	0.4192	59.17	40.83
	M14-176-8	0.9780	0.4099	58.99	41.01
	M14-176-9	1.0343	0.4261	59.71	40.29
	M14-176-10	0.9990	0.4152	59.35	40.65
	average			59.28	40.72
	uncertainty			0.23	0.23
	SD			0.32	0.32
	RSD%			0.53	0.77

melamine-m-aramid (Nomex) (sample 177)

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
90% HCOOH	177-1	0.8778	0.5645	65.16	34.84
90°C	177-2	0.8032	0.5139	64.83	35.17
1 hour	177-3	0.8452	0.5466	65.53	34.47
	177-4	0.8941	0.5744	65.10	34.90
	177-5	0.8715	0.5589	64.98	35.02
	177-6	1.0003	0.6409	64.92	35.08
	177-7	0.9771	0.6306	65.39	34.61
	177-8	1.0100	0.6482	65.03	34.97
	177-9	1.0099	0.6460	64.82	35.18
	177-10	0.9950	0.6370	64.87	35.13
	average			65.06	34.94
	uncertainty			0.00	0.00
	SD			0.24	0.24
	RSD%			0.37	0.68

method	JRC code	sample mass	residue mass	m-aramid	melamine
		g	g	%	%
50% H2SO4	177-1	0.8396	0.5619	67.80	32.20
90°C	177-2	0.8266	0.5544	67.94	32.06
1 hour	177-3	0.9946	0.6665	67.89	32.11
	177-4	0.9071	0.6040	67.46	32.54
	177-5	0.8353	0.5599	67.90	32.10
	177-6	1.0127	0.6809	68.11	31.89
	177-7	0.9998	0.6641	67.29	32.71
	177-8	0.9993	0.6690	67.82	32.18
	177-9	0.9964	0.6672	67.83	32.17
	177-10	1.0009	0.6648	67.29	32.71
	average			67.73	32.27
	uncertainty			0.20	0.20
	SD			0.28	0.28
	RSD%			0.42	0.88

melamine-p-aramid (Kevlar) (sample 178)

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
90% HCOOH	178-1	0.9217	0.4872	53.62	46.38
90°C	178-2	0.9732	0.5103	53.19	46.81
1 hour	178-3	0.8732	0.4602	53.46	46.54
	178-4	0.9885	0.5215	53.52	46.48
	178-5	0.9544	0.5031	53.47	46.53
	178-6	0.9022	0.4770	53.63	46.37
	178-7	0.9387	0.4913	53.09	46.91
	178-8	1.0129	0.5344	53.52	46.48
	178-9	0.9999	0.5316	53.93	46.07
	178-10	0.9806	0.5201	53.80	46.20
	178-11	0.7396	0.3937	53.99	46.01
	178-12	0.8749	0.4624	53.61	46.39
	178-13	0.9173	0.4850	53.63	46.37
	178-14	0.9799	0.5159	53.41	46.59
	178-15	0.8646	0.4569	53.61	46.39
	average			53.57	46.43
	uncertainty			0.13	0.13
	SD			0.24	0.24
	RSD%			0.45	0.51

method	JRC code	sample mass	residue mass	p-aramid	melamine
		g	g	%	%
50% H2SO4	178-1	0.9843	0.5233	53.93	46.07
90°C	178-2	0.9267	0.4910	53.74	46.26
1 hour	178-3	0.9400	0.4985	53.79	46.21
	178-4	0.9310	0.4952	53.95	46.05
	178-5	0.8745	0.4635	53.76	46.24
	178-6	1.0057	0.5350	53.96	46.04
	178-7	1.0501	0.5572	53.82	46.18
	178-8	0.9931	0.5274	53.87	46.13
	178-9	1.0066	0.5356	53.97	46.03
	178-10	0.9989	0.5314	53.96	46.04
	average			53.88	46.12
	uncertainty			0.06	0.06
	SD			0.09	0.09
	RSD%			0.17	0.19

Influence of HCOOH concentration on the method proposed by the applicant

melamine-cotton (sample 175)

method	JRC code	sample mass g	residue mass g	cotton %	melamine %
98-100% HCOOH	175-1	0.9998	0.4075	41.09	58.91
90°C	175-2	0.9839	0.4018	41.17	58.83
1 hour	175-3	1.0123	0.4130	41.13	58.87
	average			41.13	58.87
	uncertainty			0.10	0.10
	SD			0.04	0.04
	RSD			0.10	0.07

method	JRC code	sample mass g	residue mass g	cotton %	melamine %
93% HCOOH	175-1	0.5981	0.2426	40.90	59.10
90°C	175-2	0.4524	0.1834	40.88	59.12
1 hour	175-3	0.7122	0.2908	41.17	58.83
	average			40.98	59.02
	uncertainty			0.40	0.40
	SD			0.16	0.16
	RSD			0.40	0.28

method	JRC code	sample mass g	residue mass g	cotton %	melamine %
90% HCOOH	175-1	1.4294	0.5863	41.35	58.65
90°C	175-2	1.1217	0.4601	41.36	58.64
1 hour	175-3	0.9516	0.3882	41.13	58.87
	175-4	0.9248	0.3782	41.23	58.77
	175-5	1.0866	0.4421	41.02	58.98
	average			41.22	58.78
	uncertainty			0.18	0.18
	SD			0.14	0.14
	RSD			0.35	0.25

method	JRC code	sample mass g	residue mass g	cotton %	melamine %
87% HCOOH	175-1	0.7728	0.3177	41.45	58.55
90°C	175-2	0.5505	0.2265	41.48	58.52
1 hour	175-3	0.9020	0.3698	41.33	58.67
	average			41.42	58.58
	uncertainty			0.19	0.19
	SD			0.08	0.08
	RSD			0.19	0.13

method	JRC code	sample mass	residue mass	cotton	melamine
		g	g	%	%
85% HCOOH	175-1	0.9780	0.4185	43.13	56.87
90°C	175-2	0.9002	0.3840	43.00	57.00
1 hour	175-3	0.8613	0.3652	42.74	57.26
	175-4	0.7143	0.3050	43.04	56.96
	175-5	0.9589	0.4088	42.97	57.03
	average			42.98	57.02
	uncertainty			0.15	0.15
	SD			0.15	0.15
	RSD%			0.34	0.25

method	JRC code	sample mass	residue mass	cotton	melamine
		g	g	%	%
80% HCOOH	175-1	0.9434	0.4075	43.54	56.46
90°C	175-2	0.8927	0.3860	43.58	56.42
1 hour	175-3	1.0036	0.4326	43.45	56.55
	175-4	1.2006	0.5200	43.65	56.35
	175-5	0.9954	0.4298	43.52	56.48
	average			43.55	56.45
	uncertainty			0.08	0.08
	SD			0.08	0.08
	RSD%			0.18	0.14

Influence of temperature on the hot H2SO4 method proposed by the applicant

100% melamine (samples 136,137)

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
50% H2SO4	136-1	0.9707	0.0142	98.63	1.37
80°C	136-2	0.9649	0.0193	98.13	1.87
1 hour	136-3	0.9677	0.0144	98.61	1.39
	136-4	0.9299	0.0101	98.98	1.02
	136-5	0.9849	0.0183	98.26	1.74
	average			98.52	1.48
	uncertainty			0.42	0.42
	SD			0.34	0.34
	RSD%			0.34	22.85

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
50% H2SO4	136-1	0.9724	0.0042	99.60	0.40
85°C	136-2	1.0895	0.0001	99.99	0.01
1 hour	136-3	0.9599	0.0032	99.69	0.31
	136-4	0.9247	0.0031	99.69	0.31
	136-5	0.8867	0.0000	100.00	0.00
	average			99.79	0.21
	uncertainty			0.23	0.23
	SD			0.19	0.19
	RSD%			0.19	91.20

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
50% H2SO4	137-6	0.9253	0.0001	99.99	0.01
90°C	137-7	1.0075	0.0007	99.94	0.06
30 min	137-8	0.8709	0.0001	99.99	0.01
	137-9	0.9363	0.0004	99.96	0.04
	137-10	1.0685	0.0002	99.98	0.02
	average			99.97	0.03
	uncertainty			0.03	0.03
	SD			0.02	0.02
	RSD%			0.02	82.51

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
50% H2SO4	136-1	1.0682	0.0052	99.54	0.46
90°C	136-2	0.9626	0.0042	99.59	0.41
1 hour	136-3	0.9584	0.0058	99.43	0.57
	136-4	0.9894	0.0037	99.65	0.35
	136-5	0.9615	0.0015	99.85	0.15
	136-6	0.9885	0.0086	99.19	0.81
	136-7	1.0452	0.0066	99.41	0.59
	136-8	0.9722	0.0042	99.60	0.40
	136-9	0.9593	0.0030	99.71	0.29
	136-10	0.9777	0.0056	99.46	0.54
	average			99.54	0.46
	uncertainty			0.13	0.13
	SD			0.18	0.18
	RSD%			0.18	40.28

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
50% H2SO4	137-1	0.9385	0.0004	99.96	0.04
90°C	137-2	0.9795	0.0003	99.97	0.03
2.5 hours	137-3	1.0077	0.0009	99.92	0.08
	137-4	1.0010	0.0009	99.92	0.08
	137-5	0.8264	0.0003	99.97	0.03
	average			99.95	0.05
	uncertainty			0.03	0.03
	SD			0.03	0.03
	RSD%			0.03	50.89

Influence of temperature on the hot HCOOH method proposed by the applicant

100% melamine (samples 136,137)

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
90% HCOOH	136-1	0.9302	0.0018	99.82	0.18
88°C	136-2	0.9224	0.0014	99.86	0.14
1 hour	136-3	0.9557	0.0017	99.83	0.17
	136-4	0.9547	0.0008	99.92	0.08
	136-5	0.8520	0.0014	99.85	0.15
	average			99.86	0.14
	uncertainty			0.05	0.05
	SD			0.04	0.04
	RSD%			0.04	27.45

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
90 % HCOOH	137-1	0.7939	0.0005	99.94	0.06
90°C	137-2	0.8839	0.0003	99.97	0.03
1 hour	137-3	0.9145	0.0006	99.94	0.06
	137-4	0.9196	0.0004	99.96	0.04
	137-5	0.9641	0.0005	99.95	0.05
	136-1	0.9708	0.0005	99.95	0.05
	136-2	0.9665	0.0008	99.92	0.08
	136-3	0.9847	0.0006	99.94	0.06
	136-4	0.9861	0.0004	99.96	0.04
	136-5	0.9640	0.0010	99.90	0.10
	average			99.94	0.06
	uncertainty			0.01	0.01
	SD			0.02	0.02
	RSD%			0.02	35.02

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
90% HCOOH	136-1	0.9437	0.0013	99.87	0.13
92°C	136-2	1.0201	0.0012	99.89	0.11
1 hour	136-3	0.9937	0.0017	99.84	0.16
	136-4	0.9832	0.0012	99.89	0.11
	136-5	0.9650	0.0010	99.90	0.10
	average			99.88	0.12
	uncertainty			0.03	0.03
	SD			0.02	0.02
	RSD%			0.02	19.77

40% melamine – 60% m-aramid (Nomex) (sample 138)

method	JRC code	sample mass	residue mass	Nomex	melamine
		g	g	%	%
90% HCOOH	138-1	1.0013	0.8935	90.21	9.79
80C°	138-2	0.9896	0.8896	90.87	9.13
1 hour	138-3	0.9919	0.8946	91.17	8.83
	138-4	1.0230	0.9145	90.37	9.63
	138-5	0.9625	0.8603	90.36	9.64
	138-6	1.0316	0.9248	90.62	9.38
	138-7	1.0012	0.8980	90.67	9.33
	138-8	0.8332	0.7474	90.68	9.32
	138-9	1.0167	0.9119	90.67	9.33
	138-10	1.0813	0.9696	90.65	9.35
	average			90.63	9.37
	uncertainty			0.20	0.20
	SD			0.27	0.27
	RSD%			0.30	2.92

method	JRC code	sample mass	residue mass	Nomex	melamine
		g	g	%	%
90% HCOOH	138-1	1.0343	0.8238	80.59	19.41
85°C	138-2	1.0449	0.8256	79.95	20.05
1 hour	138-3	1.0286	0.8214	80.80	19.20
	138-4	1.0982	0.8745	80.57	19.43
	138-5	1.0522	0.8413	80.90	19.10
	138-6	0.9890	0.7890	80.72	19.28
	138-7	0.8919	0.7115	80.72	19.28
	138-8	1.0882	0.8700	80.89	19.11
	138-9	0.9419	0.7504	80.61	19.39
	138-10	1.0071	0.8025	80.63	19.37
	average			80.64	19.36
	uncertainty			0.19	0.19
	SD			0.27	0.27
	RSD%			0.33	1.39

method	JRC code	sample mass	residue mass	Nomex	melamine
		g	g	%	%
90% HCOOH	138-1	1.0754	0.6361	59.97	40.03
90°C	138-2	0.9214	0.5467	60.15	39.85
1 hour	138-3	1.0703	0.6339	60.04	39.96
	138-4	1.0772	0.6435	60.56	39.44
	138-5	1.0415	0.6153	59.89	40.11
	138-6	0.9567	0.5785	61.29	38.71
	138-7	1.0995	0.6499	59.92	40.08
	138-8	0.9645	0.5740	60.33	39.67
	138-9	1.1087	0.6634	60.66	39.34
	138-10	1.0775	0.6429	60.49	39.51
	average			60.33	39.67
	uncertainty			0.00	0.00
	SD			0.44	0.44
	RSD%			0.72	1.10

method	JRC code	sample mass	residue mass	Nomex	melamine
		g	g	%	%
90% HCOOH	138-1	0.9440	0.5665	60.83	39.17
95°C	138-2	0.9763	0.5862	60.87	39.13
1 hour	138-3	1.0055	0.6072	61.21	38.79
	138-4	1.0150	0.6153	61.45	38.55
	138-5	0.9811	0.5937	61.34	38.66
	average			61.14	38.86
	uncertainty			0.35	0.35
	SD			0.28	0.28
	RSD%			0.46	0.72

40% melamine – 60% p-aramid (Kevlar) (sample 141)

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	141-1	0.9674	0.8979	93.80	6.20
80°C	141-2	1.0352	0.9528	93.02	6.98
1 hour	141-3	0.9737	0.9024	93.66	6.34
	141-4	1.0629	0.9866	93.80	6.20
	141-5	1.0815	1.0089	94.27	5.73
	141-6	1.0817	0.9986	93.30	6.70
	141-7	1.1751	1.0873	93.51	6.49
	141-8	1.1722	1.0823	93.31	6.69
	141-9	1.1634	1.0631	92.36	7.64
	141-10	1.1404	1.0825	95.91	4.09
	141-11	1.0211	0.9485	93.87	6.13
	141-12	0.8643	0.8025	93.83	6.17
	141-13	1.0071	0.9342	93.74	6.26
	average			93.72	6.28
	uncertainty			0.49	0.49
	SD			0.81	0.81
	RSD%			0.86	12.91

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	141-1	0.9515	0.7352	78.20	21.80
85°C	141-2	0.9932	0.7682	78.28	21.72
1 hour	141-3	1.0854	0.8384	78.17	21.83
	141-4	1.0134	0.7969	79.57	20.43
	141-5	0.9737	0.7656	79.57	20.43
	141-6	0.8857	0.6865	78.44	21.56
	average			78.71	21.29
	uncertainty			0.71	0.71
	SD			0.68	0.68
	RSD%			0.86	3.17

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	141-1	1.1112	0.6742	61.50	38.50
88°C	141-2	1.0841	0.6583	61.55	38.45
1 hour	141-3	1.1050	0.6741	61.83	38.17
	141-4	1.0474	0.6318	61.15	38.85
	141-5	0.9439	0.5692	61.13	38.87
	141-6	1.0209	0.6242	61.97	38.03
	141-7	1.0274	0.6243	61.59	38.41
	141-8	1.0583	0.6382	61.13	38.87
	141-9	0.9788	0.6007	62.20	37.80
	141-10	1.0307	0.6340	62.35	37.65
	141-11	0.9919	0.6049	61.81	38.19
	141-12	1.0132	0.6209	62.11	37.89
	141-13	0.9828	0.5991	61.79	38.21
	average			61.70	38.30
	uncertainty			0.25	0.25
	SD			0.41	0.41
	RSD%			0.66	1.06

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	141-1	0.9669	0.5893	61.78	38.22
90°C	141-2	0.9923	0.6048	61.78	38.22
1 hour	141-3	0.9916	0.6008	61.42	38.58
	141-4	0.9954	0.6037	61.48	38.52
	141-5	0.9548	0.5832	61.91	38.09
	141-6	0.9987	0.6051	61.42	38.58
	141-7	1.0233	0.6186	61.28	38.72
	141-8	1.0001	0.6065	61.47	38.53
	141-9	0.9807	0.5936	61.35	38.65
	141-10	1.0478	0.6365	61.57	38.43
	average			61.54	38.46
	uncertainty			0.15	0.15
	SD			0.21	0.21
	RSD%			0.34	0.55

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	141-1	1.0612	0.6467	61.77	38.23
92°C	141-2	0.9404	0.5677	61.19	38.81
1 hour	141-3	1.0042	0.6030	60.87	39.13
	141-4	0.9246	0.5577	61.14	38.86
	141-5	1.0495	0.6357	61.40	38.60
	141-6	1.1187	0.6773	61.37	38.63
	141-7	1.0583	0.6402	61.32	38.68
	141-8	1.1388	0.6883	61.27	38.73
	141-9	1.0410	0.6269	61.04	38.96
	141-10	1.0625	0.6477	61.79	38.21
	average			61.32	38.68
	uncertainty			0.21	0.21
	SD			0.29	0.29
	RSD%			0.47	0.75

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	141-1	0.9798	0.5915	61.19	38.81
95°C	141-2	0.8806	0.5286	60.85	39.15
1 hour	141-3	1.0889	0.6565	61.11	38.89
	141-4	0.9377	0.5627	60.83	39.17
	141-5	1.0197	0.6182	61.45	38.55
	141-6	0.9628	0.5790	60.96	39.04
	141-7	0.9988	0.6043	61.33	38.67
	141-8	1.1305	0.6845	61.37	38.63
	141-9	1.0003	0.6159	62.41	37.59
	141-10	1.0128	0.6066	60.71	39.29
	average			61.22	38.78
	uncertainty			0.35	0.35
	SD			0.49	0.49
	RSD%			0.79	1.25

40% melamine – 60% p-aramid (Kevlar) (sample 142)

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	142-1	1.1445	1.0661	94.13	5.87
80°C	142-2	1.1783	1.0938	93.81	6.19
1 hour	142-3	1.1846	1.1195	95.49	4.51
	142-4	1.0389	0.9622	93.60	6.40
	142-5	1.4885	1.3736	93.26	6.74
	142-6	0.9778	0.9038	93.41	6.59
	142-7	1.0274	0.9518	93.62	6.38
	142-8	0.9209	0.8511	93.40	6.60
	142-9	1.0545	0.9771	93.64	6.36
	142-10	1.0591	0.9794	93.46	6.54
	average			93.78	6.22
	uncertainty			0.00	0.00
	SD			0.65	0.65
	RSD%			0.69	10.42

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	142-1	1.1622	1.0093	87.81	12.19
85°C	142-2	1.1813	1.0324	88.37	11.63
1 hour	142-3	1.1147	0.9624	87.30	12.70
	142-4	1.0846	0.9355	87.22	12.78
	142-5	1.2075	1.0430	87.34	12.66
	142-6	0.9457	0.8168	87.34	12.66
	142-7	0.9769	0.8412	87.08	12.92
	142-8	0.9233	0.7969	87.28	12.72
	142-9	0.9693	0.8367	87.29	12.71
	142-10	0.9549	0.8237	87.23	12.77
	average			87.42	12.58
	uncertainty			0.27	0.27
	SD			0.38	0.38
	RSD%			0.44	3.03

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	142-1	1.1388	0.6748	60.07	39.93
90°C	142-2	1.0302	0.6133	60.35	39.65
1 hour	142-3	0.9707	0.5779	60.35	39.65
	142-4	0.9684	0.5725	59.93	40.07
	142-5	1.0012	0.5913	59.87	40.13
	142-6	0.9143	0.5452	60.45	39.55
	142-7	1.0166	0.6061	60.44	39.56
	142-8	0.9527	0.5635	59.96	40.04
	142-9	1.0395	0.6164	60.11	39.89
	142-10	0.9839	0.5824	60.01	39.99
	average			60.16	39.84
	uncertainty			0.16	0.16
	SD			0.22	0.22
	RSD%			0.37	0.56

method	JRC code	sample mass	residue mass	Kevlar	melamine
		g	g	%	%
90% HCOOH	142-1	1.1896	0.7434	63.33	36.67
95°C	142-2	1.0251	0.6413	63.40	36.60
1 hour	142-3	0.9909	0.6165	63.06	36.94
	142-4	0.8769	0.5524	63.84	36.16
	142-5	0.9717	0.5987	62.45	37.55
	142-6	1.1326	0.7064	63.21	36.79
	142-7	0.9907	0.6142	62.83	37.17
	142-8	1.0084	0.6320	63.52	36.48
	142-9	1.1413	0.7090	62.96	37.04
	142-10	1.0849	0.6782	63.35	36.65
	average			63.20	36.80
	uncertainty			0.28	0.28
	SD			0.39	0.39
	RSD%			0.62	1.06

Influence of temperature on the hot NaClO method proposed by the applicant

100% melamine (sample 136)

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
6.15% NaClO	136-1	0.9567	0.9144	95.86	4.14	1.046
80°C	136-2	0.9568	0.9137	95.78	4.22	1.047
1 hour	136-3	0.9823	0.9411	96.07	3.93	1.044
	136-4	0.9678	0.9252	95.87	4.13	1.046
	136-5	0.9775	0.9376	96.17	3.83	1.043
	average			95.95	4.05	1.045
	uncertainty			0.21	0.21	0.002
	SD			0.17	0.17	0.002
	RSD%			0.17	4.08	0.18

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
6.15% NaClO	136-1	1.0143	0.9660	95.54	4.46	1.050
85°C	136-2	0.9703	0.9223	95.36	4.64	1.052
1 hour	136-3	0.9827	0.9381	95.75	4.25	1.048
	136-4	0.9647	0.9191	95.57	4.43	1.050
	136-5	0.9775	0.9293	95.38	4.62	1.052
	average			95.52	4.48	1.050
	uncertainty			0.20	0.20	0.002
	SD			0.16	0.16	0.002
	RSD%			0.16	3.51	0.18

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
6.15% NaClO	136-1	0.8907	0.8336	93.98	6.02	1.068
90°C	136-2	0.7765	0.7270	94.02	5.98	1.068
1 hour	136-3	0.7613	0.7149	94.28	5.72	1.065
	136-4	0.8402	0.7922	94.64	5.36	1.061
	136-5	0.7037	0.6606	94.25	5.75	1.065
	136-6	0.8455	0.7998	94.93	5.07	1.057
	136-7	0.9035	0.8456	93.99	6.01	1.068
	136-8	0.8432	0.7888	93.94	6.06	1.069
	136-9	1.0516	0.9947	94.93	5.07	1.057
	136-10	1.0431	0.9760	93.96	6.04	1.069
	136-11	1.0178	0.9521	93.94	6.06	1.069
	average			94.29	5.71	1.065
	uncertainty			0.27	0.27	0.003
	SD			0.40	0.40	0.005
	RSD%			0.42	6.97	0.45

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
6.15% NaClO	136-1	1.1482	0.9990	87.75	12.25	1.149
95°C	136-2	1.3095	1.1581	89.11	10.89	1.131
1 hour	136-3	1.0707	0.9526	89.62	10.38	1.124
	136-4	1.2244	1.0771	88.67	11.33	1.137
	136-5	1.1682	0.9999	86.41	13.59	1.168
	136-6	1.0962	0.9660	88.81	11.19	1.135
	136-7	0.9273	0.8182	88.92	11.08	1.133
	136-8	0.9862	0.8681	88.72	11.28	1.136
	136-9	1.1040	0.9707	88.63	11.37	1.137
	136-10	1.0119	0.8928	88.91	11.09	1.133
	average			88.55	11.45	1.138
	uncertainty			0.60	0.60	0.008
	SD			0.89	0.89	0.012
	RSD%			1.00	7.74	1.08

Influence of temperature on method 2 of Directive 96/73/EC

100% melamine (sample 136)

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
method 2	136-1	1.0368	1.0105	97.63	2.37	1.026
70°C	136-2	0.9906	0.9663	97.70	2.30	1.025
1 hour	136-3	0.9927	0.9667	97.55	2.45	1.027
	136-4	0.8657	0.8452	97.78	2.22	1.024
	136-5	1.0259	0.9989	97.54	2.46	1.027
	average			97.64	2.36	1.026
	uncertainty			0.13	0.13	0.001
	SD			0.11	0.11	0.001
	RSD%			0.11	4.45	0.11

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
method 2	136-1	1.0074	0.9744	96.93	3.07	1.034
80°C	136-2	1.1527	1.1149	96.93	3.07	1.034
1 hour	136-3	0.9598	0.9287	96.97	3.03	1.033
	136-4	0.9588	0.9262	96.82	3.18	1.035
	136-5	1.0521	1.0167	96.85	3.15	1.035
	average			96.90	3.10	1.034
	uncertainty			0.08	0.08	0.001
	SD			0.06	0.06	0.001
	RSD%			0.07	2.03	0.07

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
method 2	136-1	0.9290	0.8809	95.14	4.86	1.055
90°C	136-2	0.9004	0.8540	95.17	4.83	1.054
1 hour	136-3	0.8814	0.8353	95.10	4.90	1.055
	136-4	0.9308	0.8805	94.93	5.07	1.057
	136-5	0.8781	0.8317	95.04	4.96	1.056
	average			95.08	4.92	1.055
	uncertainty			0.12	0.12	0.001
	SD			0.09	0.09	0.001
	RSD%			0.10	1.91	0.11

Influence of temperature on method 7 of Directive 96/73/EC

100% melamine (samples 136,137)

method	JRC code	sample mass g	residue mass g	melamine %	other %	d
method 7	136-1	0.9177	0.9112	99.34	0.66	1.007
48°C	136-2	0.9540	0.9476	99.37	0.63	1.007
	136-3	1.0168	1.0085	99.24	0.76	1.008
	136-4	0.9738	0.9675	99.40	0.60	1.007
	136-5	0.9900	0.9812	99.17	0.83	1.009
	average			99.30	0.70	1.008
	uncertainty			0.10	0.10	0.001
	SD			0.10	0.10	0.001
	RSD%			0.10	13.78	0.104

method	JRC code	sample mass g	residue mass g	melamine %	other %	d
method 7	136-1	1.1359	1.1276	99.32	0.68	1.007
50°C	136-2	1.0529	1.0432	99.14	0.86	1.009
	136-3	0.912	0.9053	99.31	0.69	1.007
	136-4	1.0326	1.024	99.22	0.78	1.008
	136-5	1.0101	1.0028	99.32	0.68	1.007
	137-1	1.2328	1.2223	99.20	0.80	1.009
	137-2	1.1517	1.1430	99.29	0.71	1.008
	137-3	0.9704	0.9601	99.01	0.99	1.011
	137-4	1.0178	1.0038	98.71	1.29	1.014
	137-5	1.0092	0.9971	98.88	1.12	1.012
	average			99.14	0.86	1.009
	uncertainty			0.15	0.15	0.002
	SD			0.21	0.21	0.002
	RSD%			0.21	24.46	0.227

method	JRC code	sample mass g	residue mass g	melamine %	other %	d
method 7	136-1	0.8924	0.8835	99.07	0.93	1.010
52°C	136-2	0.9639	0.9523	98.87	1.13	1.012
	136-3	1.1255	1.1131	98.97	1.03	1.011
	136-4	1.0047	0.9974	99.32	0.68	1.007
	136-5	0.9797	0.9678	98.86	1.14	1.012
	average			99.02	0.98	1.011
	uncertainty			0.19	0.19	0.002
	SD			0.19	0.19	0.002
	RSD%			0.19	19.12	0.202

method	JRC code	sample mass g	residue mass g	melamine %	other %	d
method 7	136-1	1.3038	1.1954	92.19	7.81	1.091
55°C	136-2	0.8848	0.8119	92.26	7.74	1.090
	136-3	1.1919	1.0928	92.19	7.81	1.091
	136-4	1.0902	1.0020	92.40	7.60	1.088
	136-5	0.9409	0.8656	92.48	7.52	1.087
	average			92.30	7.70	1.089
	uncertainty			0.13	0.13	0.002
	SD			0.13	0.13	0.002
	RSD%			0.14	1.72	0.152

method	JRC code	sample mass	residue mass	melamine	other	d
		g	g	%	%	
method 7	136-1	0.9173	0.6778	75.17	24.83	1.353
60°C	136-2	1.0145	0.7516	75.36	24.64	1.350
	136-3	0.9043	0.6680	75.15	24.85	1.354
	136-4	0.6163	0.4541	74.97	25.03	1.357
	136-5	0.9338	0.6816	74.30	25.70	1.370
	average			74.99	25.01	1.357
	uncertainty			0.42	0.42	0.008
	SD			0.41	0.41	0.008
	RSD%			0.55	1.64	0.577

method	JRC code	sample mass	residue mass	melamine	other
		g	g	%	%
method 7	136-1	0.8196	0.0042	0.55	99.45
70°C	136-2	1.1453	0.0029	0.27	99.73
	136-3	1.0335	0.0014	0.14	99.86
	136-4	1.0816	0.0036	0.36	99.64
	136-5	1.0275	0.0044	0.46	99.54
	average			0.36	99.64
	uncertainty			0.16	0.16
	SD			0.16	0.16
	RSD%			44.27	0.16

European Commission

EUR 23482 EN – Joint Research Centre – Institute for Health and Consumer Protection
Title: Fibre Labelling. Melamine – Basofil.
Author(s): P. Piccinini, E. Buriova, C. Senaldi, S. Yazgan
Luxembourg: Office for Official Publications of the European Communities
2008 – 166 pp. – 21.0 x 29.7 cm
EUR – Scientific and Technical Research series – ISSN 1018-5593
ISBN 978-92-79-09577-1
DOI 10.2788/91012

Abstract
In August 2006, the European Commission's Joint Research Centre (JRC) was asked by DG Enterprise to conduct experimental work to check the validity and suitability of the testing methods proposed by Basofil for the identification, quantification and characterisation of their new fibre melamine.
Experimental results showed that microscopic analysis can be useful but is not the only technique for the identification of melamine as it can give rise to mistakes. Identification should be based on FT-IR possibly combined with Thermo Gravimetric (TG) analysis and solubility properties of the fibre.
The agreed allowance of melamine fibre was experimentally evaluated and the value of 7.00 was established.
The normal pre-treatment was shown to be applicable to the new fibre. Melamine is insoluble with all methods of the Directive 96/73/EC. The correction factors d were generally equal to 1.01, except in the case of method 3 and 10, for which d value was 1.00. Five correction factors d of melamine were confirmed through a ring trial including seventeen laboratories. The inter-laboratory trial results established the value of 1.01 as melamine correction factor for methods 3, 4, 8 and 14. The d value calculated in the ring trial for method 7 was 1.07, however the repeatability and reproducibility limits calculated were very poor and the data could not be considered as being part of a normal distribution. Consequently, it was decided that method 7 would not be considered applicable to blends containing melamine.
Three methods were proposed by the applicant and tested by the JRC for quantification purposes. For the three methods, solubility properties of melamine and other common fibres, supposed to be found in mixture with melamine, were evaluated, together with the effect of key parameters, such as contact time, temperature and concentration of reagent. The method with sodium hypochloride at 90 °C was deemed not applicable due to partial solubility of melamine, which was supposed to be insoluble.
The hot formic acid method was considered adequate and applicable in the case of binary mixtures of melamine with aramid and cotton. It was decided that the contact time could be reduced to one hour. The temperature was proved to have a strong influence on this method and it should be carefully maintained in the range 90 ± 2 °C. The hot sulphuric acid method was judged not adequate.
During the 7th meeting of the European network of national experts on textile labelling, a consensus was reached on the need to validate the new method based on hot formic acid and to confirm the correction factors d for aramid (meta and para) and cotton. The JRC organised the ring trial with the participation of seventeen European laboratories, in accordance with the rules laid down in ISO 5725 (1994). The validation was successful. The precision of the test method, which should be quoted as a percentage by mass, was expressed as repeatability and reproducibility limits. Results were 0.74 % and 1.77 %, respectively. The correction factors d for aramid and cotton were evaluated in the same context and the established values are 1.02 for both fibres.
On the basis of the experimental results and of discussions with representative experts from Member States, the definition agreed and proposed for melamine is: "fibre formed of at least 85 % by mass of cross-linked macromolecules made up of melamine derivatives".

The mission of the JRC is to provide customer-driven scientific and technical support for the conception, development, implementation and monitoring of EU policies. As a service of the European Commission, the JRC functions as a reference centre of science and technology for the Union. Close to the policy-making process, it serves the common interest of the Member States, while being independent of special interests, whether private or national.

JRC
EUROPEAN COMMISSION

LB-NA-23482-EN-C

Publications Office
Publications.eu.int